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FINAL REPORT

FOR

SEMICONDUCTOR RESISTIVE ELEMENT

This report covers the period I January 1961 to 31 December 1962

TEXAS INSTRUMENTS INCORPORATED 13500 N. CENTRAL EXPRESSWAY DALLAS, TEXAS

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NAVY DEPARTMENT BUREAU OF SHIPS ELECTRONICS DIVISION

NObsr =85406

SR-0080302, ST-9607

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ABSTRACT

THIS FINAL ENGINEERING REPORT FOR PHASE II OF THE CONTRACT COVERS A TWELVE-MONTH PERIOD. SOME BACKGROUND INFORMATION FROM PHASE II HAS BEEN INCLUDED AS REFERENCE. THE GENERAL OBJECTIVE OF PRODUCTING A LOW TEMPERATURE COEFFICIENT OF RESISTANCE (TCR) DEVICE FROM A SEMICONDUCTOR MATERIAL WAS ONLY PARTIALLY SUCCESSFUL. Typical TCR DEVICE VALUES ABOVE ROOM TEMPERATURE WERE NEVER MUCH LOWER THAN 150 PPM/°C FOR THE SILICON RESISTIVE ELEMENT INVESTIGATED. LOW TEMPERATURE TCR VALUES (AT -50°C) WERE AROUND 500 PPM/°C. LOAD-LIFE STABILITY AND RESISTANCE TO ENVIRON-MENTS (MOISTURE, OXIDATION) PROVED AS GOOD AS OR BETTER THAN THIN-FILM RESISTORS ON THE MARKET.

SPECIFIC PROBLEMS ENCOUNTERED (SUCH AS OHMIC CONTACTS, RESISTANCE ADJUST-MENT, JUNCTION EFFECTS, ETC.) AND THEIR SOLUTIONS HAVE BEEN DISCUSSED IN DETAIL. A MANUFACTURING PROCEDURE FOR PRODUCING A POLYCRYSTALLINE SILICON DEVICE HAS BEEN INCLUDED IN THIS REPORT.

A PROBLEM OF TCR SHIFT RELATED TO THE PIEZORESISTIVE EFFECT IN THE SILICON ELEMENT, THROUGH STRESS INDUCED BY THE MOLDING EPOXY, BECAME CRITICAL BUT WAS SOLVED DURING THE LAST QUARTER. DATA ON LOAD-LIFE AND ENVIRON-MENTAL TESTS WERE ACCUMULATED ON 150 FINISHED DEVICES WHICH ACCOMPANIED THIS REPORT. POTENTIAL APPLICATIONS FOR POLYCRYSTALLINE SILICON RESISTORS WERE SUGGESTED, BUT ADDITIONAL R&D WORK ON THIS APPROACH TO A LOW TC, STABLE RESISTIVE ELEMENT WAS NOT RECOMMENDED.

THE DEVELOPMENT WORK PERFORMED UNDER THIS R&D CONTRACT IS BELIEVED TO

HAVE PMPROVED THE "STATE-OF-THE-ART" OF SEMI-CONDUCTOR RESISTIVE ELEMENTS
IN GENERAL, AND IS BELIEVED TO HAVE CONTRIBUTED TO A BETTER UNDERSTANDING OF SILICON AS A RESISTIVE MATERIAL.

PURPOSE

AN ATTEMPT HAS BEEN MADE UNDER THIS CONTRACT TO DEVELOP A SEMI-CONDUCTOR RESISTIVE ELEMENT WITH A MINIMUM TEMPERATURE COEFFICIENT OF RESISTANCE (TCR) WHICH WOULD EXHIBIT GREATER RELIABILITY THAN METAL-FILM RESISTORS PRESENTLY ON THE MARKET. SPECIFIC OBJECTIVES WERE TO PRODUCE A RESISTOR WITH < 50 PPM/°C TCR (FROM -50°C TO +175°C) AND WITH LOAD-LIFE STABILITY CHARACTERISTICS COMPARABLE TO THOSE SPECIFIED BY MIL-R-10509D FOR PRECISION FIXED-FILM RESISTORS.

THE FIRST APPROACH WAS TO CONSIDER BULK SEMICONDUCTOR ELEMENTS IN ORDER TO GAIN AN ADVANTAGE OVER THE INHERENTLY UNRELIABLE NATURE OF THIN-FILM ELEMENTS. THE RESISTIVITY OF BULK-TYPE ELEMENTS WOULD NEED TO BE LARGE IN COMPARISON WITH METALS* USED IN THIN-FILM DEVICES. LIMITATIONS ON GEOMETRY OF THE ELEMENT, I.E. CROSS-SECTIONAL AREA AND EFFECTIVE LENGTH, ARE IMPOSED BY PRACTICAL CONSIDERATIONS. Unfortunately there is a LARGE VARIATION OF CONDUCTIVITY WITH CHANGING TEMPERATURE ASSOCIATED WITH HIGH RESISTIVITY IN SEMICONDUCTOR MATERIALS. THUS, IT WAS FUNDAMENTAL TO RESTRICT THE STUDY TO RELATIVELY LOW RESISTIVITY MATERIAL, WHICH, IN TURN, PREDETERMINED TO A LARGE EXTENT CONFIGURATION OF THE RESISTIVE ELEMENT.

From the resistance equation, $R = \rho(^L/A)$, it may be shown that for LOW VALUES OF " ρ " (RESISTIVITY) THE ALLOWED $^L/A$ (EFFECTIVE LENGTH

* BULK RESISTIVITY VALUES OF ALLOYS USED FOR METAL-FILM RESISTORS ARE
APPROXIMATELY 100 MICRO OHM-CM.

1. PURPOSE (CONTINUED)

OVER CROSS-SECTIONAL AREA) MUST BE LARGE TO OBTAIN REASONABLY LARGE VALUES OF RESISTANCE. CONSIDERING RESISTIVITIES IN THE RANGE OF .OOX Ω —cm, the mathematics dictates that the element geometry must be of a layer configuration for practical resistance values. The thickness of such a semiconductor resistive layer will be much greater (by at least two magnitudes) than metal-film resistive elements. In fact, it would be technically sound to assume that the bulk properties of the semiconductor material would predominate over any possible thin-film characteristics.

BASED ON THE CONCLUSIONS REGARDING TCR CHARACTERISTICS OF SEVERAL MATERIALS (TITANIA, SILITON CARBIDE, SILITON BORIDE) INVESTIGATED IN PHASE 1, IT WAS THOUGHT THAT A GREATER AMOUNT OF RESEARCH AND DEVELOPMENT WOULD BE NECESSARY TO ACHIEVE THE OBJECTIVES OF THE CONTRACT THROUGH SEMICONDUCTOR BULK-TYPE ELEMENTS THAN BY IMPROVING SEMICONDUCTOR-LAYER ELEMENTS. SILICON WAS CHOSEN FOR INVESTIGATION AS THE SEMI-CONDUCTOR RESISTIVE MATERIAL PRINCIPALLY BECAUSE A GREAT DEAL OF BACKGROUND INFORMATION ON DOPING PROCEDURES, VAPOR DEPOSETION, DAFFUSION TECHNIQUES, ETC. WAS AVAILABLE AT TEXAS INSTRUMENTS INCORPORATED. SILICON WAS CHOSEN FOR INVESTIGATION IN PREFERENCE TO GERMANTUM BECAUSE OF ITS HIGH TEMPERATURE LIMITATION AROUND 80°C, AT WHICH TEMPERATURE THE RESISTIVITY DECREASES RAPIDLY WITH FURTHER INCREASE IN TEMPERATURE. THIS IS THE "INTRINSIC" CONDUCTION REGION FOR THUS MATERIAL. SILICON'S INTRINSIC REGION FOR THE HIGHLY DOPED (>1018 ATOMS/CC OF IMPURITY) CONCENTRATION DOES NOT BECOME PREDOMINANT UNTIL ABOUT 500°C.

1. PURPOSE (CONTINUED)

ا ا

A. DISCUSSION OF IMPURITY CONDUCTION MECHANISMS

AT THIS POINT IT WOULD BE APPROPRIATE TO DISCUSS THE ELECTRICAL BEHAVIOR OF SILICON IN RELATION TO TEMPERATURE. FIGURE 1 IS A FAMILY OF RESISTIVITY TEMPERATURE CURVES FOR "N-TYPE" SILICON OF VARYING IMPURITY CONCENTRATION. THESE CURVES WERE PLOTTED FROM CALCULATED VALUES OF RESISTIVITY FOR VARIOUS TEMPERATURES AT AN ASSUMED ACTIVATION ENERGY FOR AN "N-TYPE" IMPURITY. THE TEMPERATURE RANGE OF INTEREST HAS BEEN OUTLINED ON THE GRAPHS. WITHIN THIS RANGE IT CAN BE SEEN THAT ONLY THE HIGHLY DOPED (1018 - 1020 AT./CC) MATERIAL EXHIBITS A MINIMUM CHANGE OF RESISTIVITY WITH TEMPERATURE. THIS MINIMUM RESISTIVITY CHANGE WITH TEMPERATURE OF HIGH IMPURITY CONCENTRATION IS RELATED TO CARRIER MOBILITY AS SHOWN IN FIGURE 2.

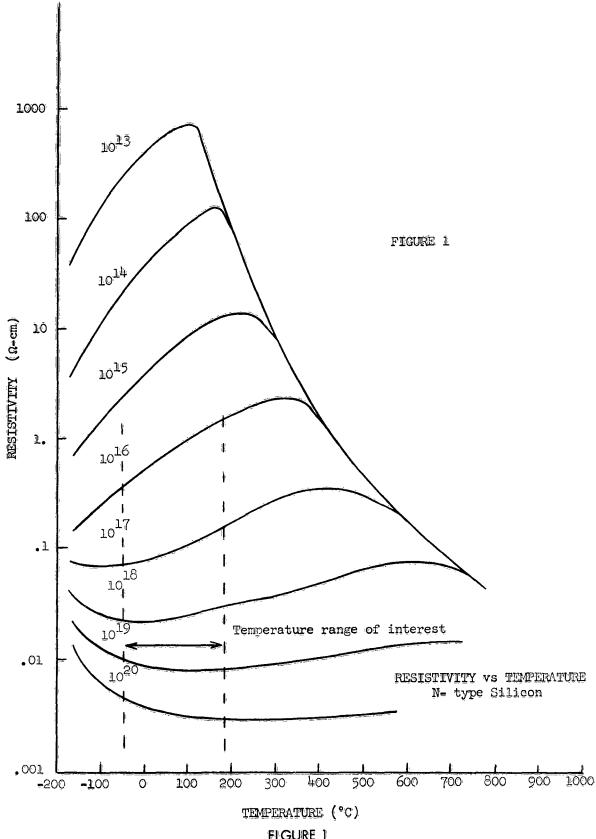
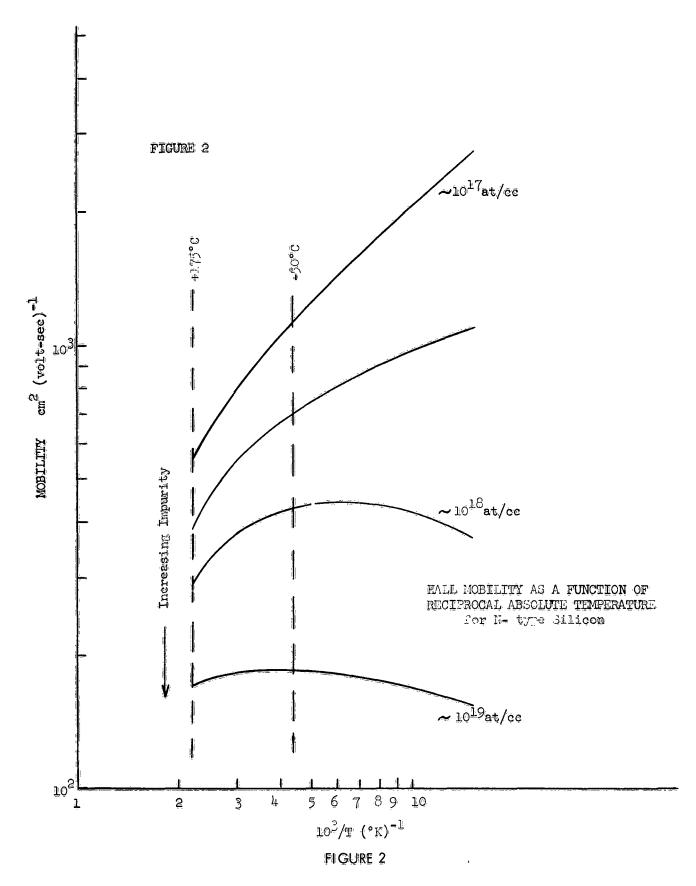


FIGURE 1



A. DISCUSSION OF IMPURITY CONDUCTION MECHANISMS (CONTID)

FIGURE 3 IS A HYPOTHETICAL CURVE, FOR "N-TYPE" OR "P-TYPE"

MATERIAL, WHICH IS AN ENLARGEMENT OF THAT SEGMENT OF THE OVER

ALL CURVE WITHIN THE TEMPERATURE RANGE AND IMPURITY LEVEL

OF INTEREST. THE DISCUSSION WHICH FOLLOWS WILL EXPLAIN IN

GREATER DETAIL THE CONDUCTION MECHANISMS WHICH TAKE PLAGE

IN THIS "EXTRINSIC" OR IMPURITY-DEPENDENT REGION.

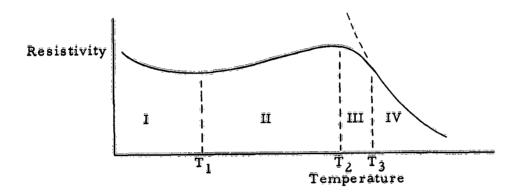


Fig. 3

THERE ARE FOUR GENERAL REGIONS INTO WHICH THE RESISTIVITY-TEMPERATURE CURVE MAY BE DIVIDED FOR PURPOSES OF DISCUSSION. IN EACH REGION THE TEMPERATURE COEFFICIENT IS DETERMINED BY CONDUCTION MECHANISMS WHICH MAY BE DESCRIBED IN FAIRLY SIMPLE TERMS.

AT SOME ARBITRARILY LOW TEMPERATURE, THE RESISTIVITY IS

DECREASING AS TEMPERATURE IS INCREASED AS A RESULT OF IMPURITIES

BEING CONTINUOUSLY IONIZED. MORE CARRIERS ARE THUS MADE AVAIL—

ABLE FOR CONDUCTION. AS TEMPERATURE T₁ IS REACHED, THE NEGATIVE

SLOPE OF THE CURVE BECOMES LESS AND THEN GOES POSITIVE BECAUSE

ALL THE IMPURITIES HAVE BEEN IONIZED AND THE MOBILITY IS BEING

A. DISCUSSION OF IMPURITY CONDUCTION MECHANISMS (CONT'D)

DECREASED DUE TO LATTICE VIBRATION OF THE CRYSTAL.

The temperature T_1 , at which this occurs is determined for a GIVEN SEMICONDUCTOR BY TWO THINGS: (1) THE DOPING LEVEL, (2) THE ACTIVATION ENERGY OF THE IMPURITY. IN GENERAL, AS THE DOPING LEVEL INGREASES, THE TEMPERATURE T1 AT WHICH ALL IMPURITY ATOMS ARE IONIZED WILL INGREASE. FOR A GIVEN IMPURITY CONCEN-TRATION, TI WILL ALSO INGREASE AS THE ACTIVATION ENERGY OF THE impurity increases. The reason for the dependence of \mathbb{T}_1 on ACTIVATION ENERGY IS THAT BEFORE AN IMPURITY ATOM CAN BE FON IZED AND AID IN CONDUCTION, IT MUST ACQUIRE SUFFICIENT ENERGY FROM SOME SOURCE. THIS ENERGY IS KNOWN AS THE ACTIVATION ENERGY OF THE IMPURITY. GALLIUM, FOR INSTANÇE, HAS A HIGHER ACTIVATION ENERGY THAN BORON, BOTH OF WHICH ARE USED TO DOPE SILICON TO MAKE IT PETYPE. IF TWO SILICON SAMPLES ARE TAKEN, ONE DOPED WITH GA TO 5 x 1018 AND ANOTHER DOPED WITH B TO 5 x 1018, T1 WILL BE HIGHER FOR THE GA-DOPED SAMPLE. THE THERMAL ENERGY GIVEN UP TO THE SEMICONDUCTOR IONIZES THE IMPURITIES, HENCE A HIGHER AMOUNT OF THERMAL ENERGY IS REQUIRED TO IONIZE A GA IMPURITY ATOM THAN IS REQUIRED FOR A B IMPURITY ATOM. SINCE THERMAL ENERGY IS PRO-PORTIONAL TO TEMPERATURE, T1 INCREASES AS ACTIVATION ENERGY INCREASES!

At T_1 all impurity atoms are considered fonezed. Since a constant number of carriers are present, the resestivity will increase as

A. DISCUSSION OF IMPURITY CONDUCTION MECHANISMS (CONT'D)

THE MOBILITY IS DECREASED DUE TO LATTICE VIBRATIONS OF THE CRYSTAL. THE MOBILITY CAN BE THOUGHT OF AS THE AVERAGE DRIFT VELOCITY WHICH A CARRIER (OR THE WAVE ASSOCIATED WITH 1T) ACQUIRES IN THE DIRECTION OF AN APPLIED ELECTRIC FIELD OF ONE VOLT PER CENTIMETER. THE LATTICE VIBRATIONS DECREASE THE MEAN FREE PATH OF THE WAVES ASSOCIATED WITH THESE CARRIERS AND HENCE DECREASE THE MOBILITY.

AS THE TEMPERATURE IS INCREASED FROM T₁, CARRIERS FROM THE SEMIGONDUCTOR ATOMS ARE ACTIVATED DUE TO THE INCREASED THERMAL ENERGY AVAILABLE. THE IMPURITY CONDUCTION DOMINATES UNTIL THE NUMBER OF CARRIERS FROM THE SEMICONDUCTOR ATOMS APPROACHES THE NUMBER OF CARRIERS FROM THE IONIZED IMPURITIES. THIS OCCURS IN THE REGION OF T₂. As more and more carriers are activated from the semiconductor atoms, the resistivity starts to drop since the number of carriers is increasing at a faster rate than the mobility is decreasing due to lattice vibrations. At some temperature, T₃, the point is reached where the semiconductor goes intrinsic. This means that above this temperature the temperature coefficient of resistance is highly negative and dependent only on the semiconductor band gap. The temperature T₃, at which the material becomes intrinsic is dependent on the Doping Level and doping type as seen from the preceding discussion.

I. PURPOSE (CONTINUED)

B. APPROACHES TO DEVELOPMENT OF LOW TER ELEMENT

IN GENERAL, THREE APPROACHES WERE CONSIDERED IN PHASE II FOR DEVELOPING THE SILICON LAYER ELEMENT. THE FIRST OF THE TECHNIQUES INVESTIGATED WAS DIFFUSION, WHERE THE RESISTIVE LAYER IS FORMED BY DIFFUSING IMPURITIES INTO HIGH RESISTIVITY SINGLE-CRYSTAL SUBSTRATES. THE OTHER TWO APPROACHES WERE VAPOR DEPOSITION TECHNIQUES FORMING SINGLE-CRYSTAL (EPITAXIAL) ELEMENTS OR POLYCRYSTALLINE LAYERS DEPOSITED ON CERAMIC SUBSTRATES.

SOME OF THE ADVANTAGES AND DISADVANTAGES OF THE THREE APPROACHES MAY BE REITERATED.

ADVANTAGES

1. DIFFUSED

UHTOSED

- A.) THE LAYER IS AN INTEGRAL PART OF THE SUBSTRATE, I.E., IT IS A MONOLITHIC STRUCTURE.
- B.) SHEET RESISTANCE MAY BE RAISED B
 TO A REASONABLE MAGNITUDE (500
 OHMS PER SQUARE) WITHOUT ENCOUNTERING PROBLEMS ASSOCIATED WITH
 THIN-FILM ELEMENTS. THIS GOES BACK
 TO THE FACT THAT THE RESISTIVE
 LAYER IS PHYSICALLY THE SAME AS C
 THE SUBSTRATE.

DISADVANTAGES

- A.) Doping to an exact concentration (e.g., 4.5 x 1018 at./cc) by diffusion might not be easily produced.
- B.) JUNCTION CONFIGURATIONS ARE SENSITIVE TO SURFACE CONTAMI= NATION WHICH OFTEN CAUSES CURRENT LEAKAGE ACROSS THE JUNCTION.
- SINGLE-CRYSTAL SILICON SUB-STRATE WOULD BE MORE EXPEN-SIVE THAN GERAMIC.

2. EPITAXIAL (VAPOR DEPOSITED ON SINGLE CRYSTAL SUBSTRATE)

- A.) THE LAYER IS AN INTEGRAL PART OF THE SUBSTRATE, I.E., IT IS A MONOLITHIC STRUCTURE.
- A.) JUNCTION CONFIGURATIONS ARE SENSITIVE TO SURFACE CONTAMINATION WHICH SOMETIMES CAUSES CURRENT LEAKAGE ACROSS THE JUNCTION.

2. EPITAXIAL (VAPOR DEPOSITED ON SINGLE CRYSTAL SUBSTRATE) (CONT'D)

- B.) SHEET RESISTANCE MAY BE RAISED B.) ADJUSTMENT OF RESISTANCE
 TO A REASONABLE MAGNITUDE (500 VALUE WILL BE DIFFICULT
 OHMS PER SQUARE) WITHOUT ENCOUNTER: (ETCHING TO A PRESCRIBED GEOME =
 ING PROBLEMS ASSOCIATED WITH THIN TRY OR ELECTRON BEAM CUTTING
 FILM ELEMENTS. THIS GOES BACK TO ARE TWO POSSIBLE METHODS).
 THE FACT THAT THE RESISTIVE LAYER
 IS PHYSICALLY THE SAME AS THE SUBSTRATE.
- C.) DISTRIBUTION OF IMPURITY ATOMS CLOSELY APPROXIMATES THAT OF CRYSTALS GROWN FROM MELT, HENCE A UNIFORMITY WITHIN THE LAYER IS OBTAINED. THIS MEANS THAT THE ELEMENT COULD BE ETCHED TO REDUCE LAYER THICKNESS (RAISING SHEET RESISTANCE) YET RETAIN THE SAME TOR CHARACTERISTICS.
- G.) SINGLE GRYSTAL SILICON SUB= STRATE WOULD BE MORE EXPENS SIVE THAN CERAMIC:

3. POLYCRYSTALLINE (VAPOR DEPOSITED ON CERAMIC SUBSTRATE)

- A.) Á THICK-LAYER HAS LESS CHANCE FOR DEVELOPING HÓT SPOTS WHICH COULD GAUSE GATASTROPHIC FAILURE.
- B.) THERMAL CONDUCTIVITY OF THE CERAMIC SUBSTRATE IS GREATER THAN SILICON, HENCE WOULD SHOW GREATER DISSIPATION OF POWER.
- C.) THE CERAMIC MATERIAL IS A LESS EXPENSIVE SUBSTRATE AND ALLOWS MORE FLEXIBILITY IN DESIGN.
- D.) RESISTANCE ADJUSTMENT IS MORE PRACTICAL.

- A.) NOT MONOLITHIC WITH SUB-STRATE. SILICON AND ALUMINA CERAMIC HAVE DIFFERING THERMAL EXPANSION COEFFICIENTS (~1:2).
- B.) POLYCRYSTALLINE LAYER WOULD NOT BE AS EASILY CONTROLLED FROM A RESISTIVITY STANDPOINT AS WOULD EPITAXIAL.
- C.) THE MINIMUM LAYER THICKNESS
 IS LIMITED FOR PRACTICAL
 REASONS TO GREATER THICKNESSES
 THAN EITHER DIFFUSED OR EPITAXIAL; THIS MEANS A LOWER
 POSSIBLE SHEET RESISTIVITY.

THE MAJOR EFFORT OF THE DEVELOPMENT WORK HAS BEEN WITH THE VAPORDEPOSITION APPROACH. EARLY IN THE SECOND PHASE OF THE WORK IT
BECAME APPARENT THAT THE DIFFUSED-LAYER APPROACH HAD MANY INHERENT
PROBLEMS AND LIMITATIONS. THE FOREMOST OF THESE WAS THE JUNCTION
EFFECT AND ITS ASSOCIATED TEMPERATURE DEPENDENCY, I.E., THE PROBLEM
OF GURRENT LEAKAGE ACROSS THE P-N JUNCTION. THE ELECTRICAL ISOLATION

B. APPROACHES TO DEVELOPMENT OF LOW TER ELEMENT (CONT'D)

OF THE MORE CONDUCTIVE LAYER FROM ITS SUBSTRATE IS DEPENDENT UPON THE EFFECT IVENESS OF THE JUNCTION TO PREVENT CURRENT PASSAGE THROUGH OR ACROSS THE SURFACE OF THE JUNCTION. CONDUCTION ACROSS THE SURFACE WHICH EXPOSES THE LAYER-SUBSTRATE JUNCTION IS GREATLY INFLUENCED BY CLEANLINESS. CONTAMINATION REMAINING ON THE SURFACE OF P-N JUNCTIONS IS ONE OF THE MOST CRITICAL PROBLEMS IN THE FABRICATION OF SEMICONDUCTOR DEVICES. THE EFFECT OF JUNCTION LEAKAGE OF THE HIGH TEMPERATURES (ABOVE 100°C) SEVERELY AFFECTED THE TCR AT THE DIFFUSED ELEMENTS AND WOULD HAVE LIMITED THEIR USEFULNESS AT THESE ELEVATED TEMPERATURES. THE INABILITY TO REPRODUCE THE EXACT DOPING LEVEL OF THE DIFFUSED LAYER (HENCE ITS TCR CHARACTERISTICS) WAS ANOTHER MAJOR PROBLEM WITH THIS APPROACH.

VAPOR DEPOSITION BY PYROLYTIC REDUCTION OF A SILICON HALIDE WAS A MUCH MORE PRACTICAL APPROACH. THICKNESS OF THE SILICON LAYER AND CONCENTRATION OF THE IMPURITY (BORON FOR P-TYPE, PHOSPHORUS FOR N=TYPE) WAS MORE EASILY CONTROLLED THAN BY DIFFUSION. POLYCRYSTALLINE LAYERS GAVE INDICATION AT THE EARLY STAGES OF DEVELOPMENT OF HAVING MORE PROMISING TCR CHARACTERISTICS THAN THE SINGLE CRYSTAL (EPITAXIAL) MATERIAL. THERE WAS NOT THE PROBLEM OF JUNCTION EFFECTS WITH SILICON DEPOSITED ON INSULATING SUBSTRATES AND OHMIC CONTACTS TO THE ELEMENT COULD BE MADE MORE STABLE (MECHANICALLY).

PRESENTED UNDER THE DETAILED FACTUAL DATA SECTION OF THIS REPORT WILL BE A DISCUSSION OF MORE SPECIFIC PROBLEMS ENCOUNTERED IN THE DEVELOPMENT OF THE SILICON LAYER ELEMENT.

1. PURPOSE (CONTINUED)

C. CHEMISTRY OF VAPOR DEPOSITION

AS IS WELL KNOWN, SILICON CAN BE PRODUCED BY THE HIGH TEMPERATURE REDUCTION OF SILICON HALIDES BY HYDROGEN. THE SIMPLIFIED EQUATION IS SHOWN BELOW:

(1) SICL $_{\downarrow}(\bar{g})$ + $2H_{\bar{Z}}(g)$ \longrightarrow SI(\bar{s}) + $4H\bar{C}L(g)$ In a kinetic system such as the system used in this study, the reaction is complicated by several side reactions. The two most prevalent side reactions are the formation of chlorosilanes (SIH $_{X}CL(1+x)$) and chlorosilane polymers $H(SIH_{X}CL_{2+x})_{Y}H$.

THE FORMATION OF CHLOROSILANES CAUSES NO DIFFICULTY IN THE FORMA-TION OF SILICON DEPOSITS SINCE THE CHLOROSILANES ARE GASEOUS IN THE REACTION SYSTEM AND ARE READILY SWEPT FROM THE REACTION ZONE.

THE FORMATION OF CHLOROSILANE POLYMERS, HOWEVER, IS DETRIMENTAL TO THE FORMATION OF SILICON DEPOSITS SINCE THE POLYMERS MAY CODEPOSIT WITH THE SILICON AND CONTAMINATE THE DEPOSITED SILICON LAYER.

BOTH SIDE REACTIONS CAN BE CONTROLLED BY A JUDICIOUS CHOICE OF REACTION CONDITIONS. AT TEMPERATURES BELOW APPROXIMATELY 1050°C, POLYMERIC CHLOROSILANES ARE READILY FORMED. ABOVE 1050°C POLYMER FORMATION IS REDUCED TO A NON-DETECTABLE LEVEL IN THE SILICON DEPOSIT. THEREFORE, IF THE SILICON DEPOSITION IS CARRIED OUT AT TEMPERATURES IN EXCESS OF 1100°C, HIGH QUALITY SILICON DEPOSITS

C. CHEMISTRY OF VAPOR DEPOSITION (CONT'D)

FREE OF POLYMERIC CONTAMINATION ARE FORMED.

ACCORDING TO EQUATION (1) ABOVE, A MOLAR RATIO OF 2:1 OF HYDROGEN TO SILICON TETRACHLORIDE IS REQUIRED FOR THIS REACTION. A MOLAR RATIO LESS THAN 2:1 WOULD, OF COURSE, HINDER THE FORMATION OF SILICON AND TEND TO PRODUCE UNDESIRABLE BY=PRODUCTS. A MOLAR RATIO OF H2:SICLL GREATER THAN 2:1 WOULD BE EXPECTED TO "FORCE" THE REACTION TO THE RIGHT FAVORING THE FORMATION OF SI(s).

ALTHOUGH THE HIGHER MOLAR RATIOS OF H2:SICLL MIGHT BE EXPECTED TO PROMOTE THE FORMATION OF SILANE (SIHL), THIS POSSIBILITY CAUSES NO PROBLEMS. If SILANE WERE FORMED IT IS A GASEOUS MATERIAL AND WOULD BE SWEPT OUT OF THE REACTOR IN THE FLOWING GAS SYSTEM.

ALSO, SILANE AND ITS HIGHER HOMOLOGS (E.G., SI2H6, SI3H8, ETC.)

ARE THERMALLY UNSTABLE AND WOULD DECOMPOSE AT THE TEMPERATURES ENCOUNTERED IN THE REACTION ZONE TO FORM SILICON AND HYDROGEN.

IN PRACTICE A MOLAR RATIO OF HYDROGEN TO SILICON TETRACHLORIDE GREATER THAN TWENTY (20) IS EMPLOYED. NORMALLY, A SILICON TETRACHLORIDE CONCENTRATION BETWEEN ONE (1) AND FOUR (4) MOLE PER CENT IN HYDROGEN IS USED.

Sample calculation of impurity doping level for SiCly:

If the desired impurity level is 5.0 x 10¹⁹ at./cc

P in Si, (see figure 4), this would be equivalent to approximately .002 ohms-cm material.

C. CHEMISTRY OF VAPOR DEPOSITION (CONT'D)

S1:
$$\frac{2.33 \text{ G/cc}}{28.09 \text{ G/G-ATOM}} \times 6.02 \times 10^{23} \text{ AT./G-ATOM} = 5.00 \times 10^{22} \text{ AT./cc}$$

$$\frac{5.0 \times 10^{19} \text{AT./cc} (P)}{5.0 \times 10^{22} \text{ AT./cc} (S1)} \times 100 = .10 \text{ MOLE \% P IN S1}$$

SICLL:
$$\frac{1.48 \text{ G/cc}}{169.89 \text{ G/MOLE}} \times 6.02 \times 10^{23} \text{ MOLECULES/MOLE} =$$

5.24 x 10²¹ MOLECULES/CC OR 5.24 x 10²⁴ MOLECULES/LITER.

PCL3 NEEDED TO DOPE SICL4 TO THE PREDETERMINED LEVEL .10 x 10^{-2} x 5.24 x 10^{24} MOLECULES/LITER = 5.24 x 10^{21} MOLECULES OF PCL3 .

PCL3
$$\frac{1.57 \text{ G/cc}}{137.39 \text{ G/MOLE}} \times 6.02 \times 10^{23} \text{ MOLECULES/MOLE} = 6.88 \times 10^{21} \text{ MOLECULES/cc.}$$

0.76 CC OF PCL3/LITER OF SICL4.

THIS CALCULATION ASSUMES THAT THE MOLAR RATIO OF PCL3 IN SICLL IN THE FEED MIXTURE WILL BE DUPLICATED AS A P IN SI RATIO IN THE PYROLYTIC DEPOSIT.

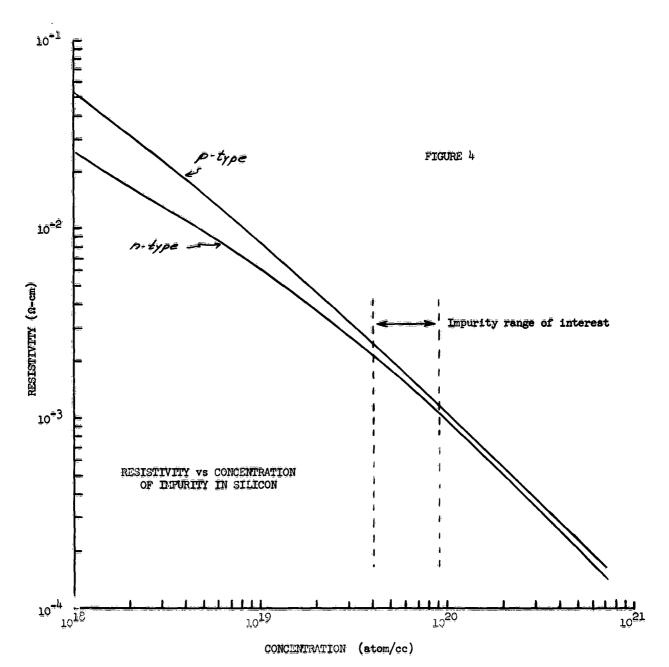


FIGURE 4

11. GENERAL FACTUAL DATA

A. IDENTIFICATION OF TECHNICAL CONTRIBUTORS

Ēng inēcrė	Mán Hours, Last Quarter
R. W. WESTBROOK	555
R. A. Rog⊎ES	400
B. L. KENNIMER	212 140
B, G. CARBAJAL	140
TECHNICIANS	
J. K. HOCKER	382
<u> Assemblers</u>	
N. Ř. ŘICHARDSON	232
W. B. HALDEMAN	169
J. A. Ďaviš	140
C. M. SHOTWELL	133

B. REFERENCES

FIRST, SECOND (FINAL FOR PHASE I), THIRD, FOURTH, AND FIFTH QUARTERLY REPORTS FOR THIS CONTRACT:

- Backenstoss, G., "Evaluation of Surface Concentration of Diffused Layers in Silicon", Bell Systems Technical Journal, 37 699-718 (1958)
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- SMITH, C. S., "PIEZORESISTANCE EFFECT IN GERMANIUM AND SILICON",
 PHYSICS REVIEW, 94, 1 (1954)

II. GENERAL FACTUAL DATA

C. MEASUREMENT PROCEDURES

MIL-R-10509D, CHARACTERISTIC C, HAS BEEN USED AS A GUIDE IN TESTING FINISHED DEVICES. FOR EVALUATION OF THE RESISTIVE ELEMENT THE FOLLOWING SPECIAL EQUIPMENT WAS USED:

TEST

- 1. SHEET RESISTANCE
- 2. THICKNESS OF LAYER

 NOTE: AN EXAMPLE OF A SILICON

 LAYER PHOTOGRAPHED UNDER SODIUM

 LIGHT FOR THICKNESS MEASURE =

 MENT MAY BE SEEN IN THE

 SUPPLEMENTARY DATA SECTION.
- 3. TCR

EQUIPMENT

FOUR-POINT PROBE TEST SET,

LAPPING APPARATUS, MICROSCOPE

WITH CAMERA ATTACHMENT, SOPIUM

LIGHT SOURCE.

SILIGONE OIL BATHS, TEMPERA-TURE CONTROLS, CHAMBER FOR LOW TEMPERATURES.

III. DETAILED FACTUAL DATA

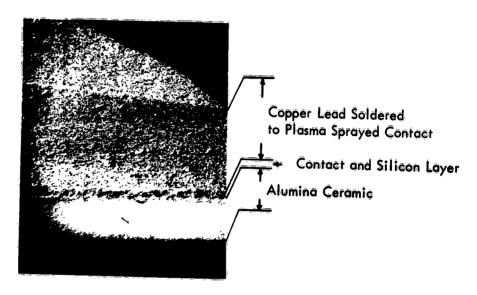
A. SPECIFIC PROBLEM AREAS

1. OHMIC CONTACTS:

PERHAPS THE MOST CRITICAL PROBLEM TO BE SOLVED IN THE DEVELOPMENT OF THE SILICON RESISTIVE ELEMENT, OTHER THAN THE FUNDAMENTAL PROBLEM OF MINIMIZING TCR, WAS THAT OF PROVIDING ELECTRICALLY AND MECHANICALLY STABLE CONTACTS. MOST OF THE VARIATION IN RESISTANCE UNDER LOAD CONDITIONS RELATED TO INSTABILITY OF THE OHMIC CONTACT TO THE SILICON ELEMENT.

THREE METHODS FOR MAKING OHMIC CONTACT WERE EVALUATED; NICKEL PLATING, GOLD AND PALLADIUM EVAPORATION, AND PLASMA SPRAYING OF ALUMINUM AND COPPER. OF THESE, THE PLASMA-SPRAYED AL-CU AND SOLDERED LEAD ATTACHMENT PROVED TO BE THE MOST RELIABLE.

THE OTHER TWO METHODS, PLATING AND EVAPORATION, RESULTED IN OHMIC CONTACTS BUT DID NOT HAVE THE NECESSARY MECHANICAL STRENGTH FOR GOOD STABILITY. LEAD WIRES ARE NORMALLY BALL-BONDED, A THERMOCOMPRESSION-TYPE BOND, TO THE EVAPORATED METAL AREAS. THIS TECHNIQUE IS USED FOR MAKING OHMIC CONTACT TO SILICON NETWORK DEVICES BUT WAS NOT SUFFICIENTLY SUBSTANTIAL FOR THE HIGHER POWER REQUIREMENTS OF A DISCRETE RESISTOR. LOAD LIFE DATA AND NOISE MEASUREMENTS WERE USED TO EVALUATE THE CONTACT METHODS (SEE SUPPLEMENTARY DATA SECTION). A MICROPHOTOGRAPH OF A CROSS-SECTION THROUGH THE PLASMA-SPRAYED SOLDER CONTACT MAY BE SEEN IN FIGURE 5.



Cross-Section of Contact Area Polycrystalline Silicon on Ceramic (50 X)

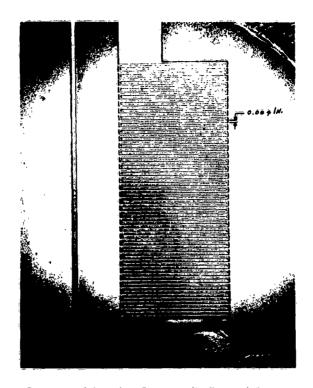
FIGURE 5

A. SPECIFIC PROBLEM AREAS

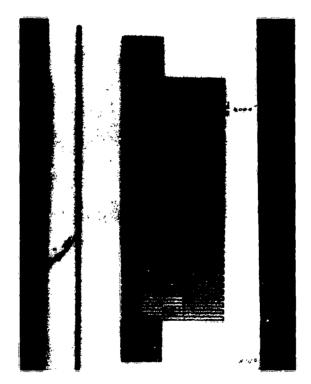
2. RESISTANCE ADJUSTMENT:

THE DEVELOPMENT OF THE SEMICONDUCTOR ELEMENT WAS TO BE DIRECTED TOWARD A MORE RELIABLE METHOD OF ADJUSTING THE VALUE OF RESISTANCE THAN BY PURELY MECHANICAL MEANS (SUCH AS SPIRALLING WITH AN ABRASIVE WHEEL) WHICH IS STANDARD PRACTICE FOR CARBON AND METAL FILM RESISTORS. TWO TECHNIQUES FOR ADJUSTING THE RESISTANCE OF THE SILICON ELEMENT WERE INVESTIGATED. THE FIRST WAS A STANDARD TECHNIQUE EMPLOYED IN MOST SEMICONDUCTOR DEVICE FABRICATION WORK; I.E., MASKING AND ETCHING. THE SECOND WAS THE MORE NOVEL APPROACH OF ELECTRON BEAM SCRIBING.

THE PHOTO-MASKING AND ETCHING TECHNIQUE WAS DESCRIBED IN DETAIL
IN PREVIOUS REPORTS. TO PRODUCE A PREDETERMINED RESISTANCE
VALUE REQUIRED A PRESCRIBED PATTERN ON THE PHOTOGRAPHIC MASK.
CHEMICAL ETCHING SIMPLY REMOVES THE SILICON WHICH HAS NOT BEEN
PROTECTED BY THE EXPOSED AND DEVELOPED RESIN (KMER, KODAK
METAL ETCH RESIST). KMER IS A PHOTO-SENSITIVE RESIN WHICH,
AFTER POLYMERIZATION, HAS EXCELLENT ACID RESISTANCE. USING
THIS TECHNIQUE IT WAS POSSIBLE TO OBTAIN PATTERNS WITH LINES
AND SPACINGS AS NARROW AS 2 MILS (.002") WITH GOOD DEFINITION.
BASE VALUES OF APPROXIMATELY 50 OHMS COULD BE ETCHED TO VALUES
OF 100K AND ABOVE. (SEE MICROPHOTOGRAPHS OF ETCHED PATTERNS ON
SINGLE-CRYSTAL AND POLYCRYSTALLINE SILICON IN FIGURES 6 AND 7
RESPECTIVELY.)



Etched Pattern of Single (Epitaxial) Crystal Silicon Layer FIGURE 6



Etched Pattern of Polycrystalline Silicon Layer FIGURE 7

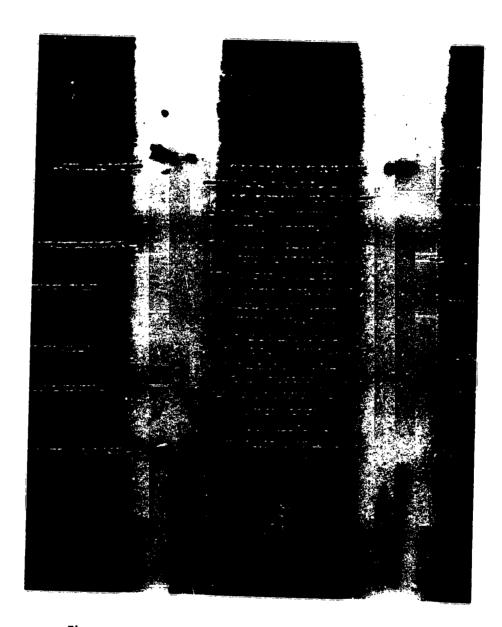
A. SPECIFIC PROBLEM AREAS (CONT'D)

ELECTRON BEAM SCRIBING WAS DONE WITH A MACHINE BUILT BY THE
ZEISS COMPANY IN GERMANY. THE TECHNIQUE COMBINES MELTING AND
EVAPORATION BY A HIGH-INTENSITY ELECTRON BEAM WHICH CAN BE
FOCUSED IN A CONTROLLABLE MANNER. THE EQUIPMENT ALLOWS THE
SUBSTRATE TO BE MANEUVERED FOR SCRIBING (OR WELDING) PRE=
DETERMINED PATTERNS. BOTH SINGLE AND POLYCRYSTALLINE ELEMENTS
WERE SCRIBED BY THIS TECHNIQUE TO LENGTHEN AND NARROW THE
RESISTANCE PATH, THUS INCREASING THE RESISTANCE VALUE OF THE
ELEMENT. FIGURE 8 SHOWS AN EXAMPLE OF SCRIBING POLYCRYSTALLINE
SILICON (DEPOSITED ON AN ALUMINA CERAMIC SUBSTRATE).

OF THE TWO METHODS, PHOTO MASKING AND ETCHING WAS EMPLOYED AS
THE PRINCIPAL MEANS OF INCREASING THE RESISTANCE ABOVE BASE
VALUES, SINCE THE TECHNIQUE WAS ALREADY KNOWN AND PRACTICED IN
THE IMMEDIATE LABORATORY AREA. THE POTENTIAL ADVANTAGE OF
ELECTRON BEAM SCRIBING AS A METHOD OF ADJUSTING THE RESISTANCE
WHILE MONITORING THE VALUE WAS NOT OVERLOOKED. THE ZEISS ELECTRON
BEAM EQUIPMENT WAS A RESEARCH MODEL WHICH WAS NOT READILY AVAILE
ABLE. THE SCRIBING OF ANY LARGE NUMBER OF ELEMENTS WOULD NOT
HAVE BEEN AS PRACTICAL AS THE ETCHING TECHNIQUE.

3. INCREASED SHEET RESISTANCE:

ONE SERIOUS LIMITATION OF THE SILIGON RESISTIVE ELEMENT WAS THE LOW RESISTIVITY NECESSARY FOR LOW TCR, WHICH IN TURN YIELDED LOW SHEET RESISTANCE OF THE DEPOSITED LAYER. BULK RESISTIVITY OF



Electron-Beam Scribed Polycrystalline Silicon Layer FIGURE 8

3. INCREASED SHEET RESISTANCE (CONT'D)

material found to be optimum for good TCR characteristics was approximately .002 ohm=cm. Sheet resistance values for thicknesses of about three microns (approx .12 mils) was about eight (8) Ω/sq on single crystal (epitaxial) layers. The polycrystalline material of comparable thickness gave higher sheet resistance values (approx 20 Ω/sq) for the same doping level.

ATTEMPTS WERE MADE TO INCREASE THE SHEET RESISTANCE OF DIFFUSED LAYER ELEMENTS BY MORE SHALLOW DIFFUSIONS INTO THE SILICON SUBSTRATE. VALUES AS HIGH AS $300~\Omega/\text{sq}$ were obtained on Diffused elements, but the uncertainty of repeating these results coupled with generally unstable resistance values made these attempts unfruitful.

THE PROBLEM OF INCREASING THE SHEET RESISTANCE OF DEPOSITED SILICON LAYERS WAS NEVER SATISFACTORILY RESOLVED. THE QUESTION STILL UNANSWERED IS, WHAT MINIMUM THICKNESS OF SILICON MAY BE DEPOSITED WITHOUT SACRIFICING RELIABILITY OF THE RESISTIVE ELEMENT. IMPROVED STABILITY OF THE SEMICONDUCTOR LAYER ELEMENTS IS BASED ON GREATER THICKNESS OF THE SILICON RELATIVE TO METAL OR CARBON FILM ELEMENTS. TO DECREASE THICKNESS WOULD PRESUMABLY REDUCE THE RELIABILITY OF THE ELEMENT, SINCE THE MAGNITUDE OF THICKNESS WOULD APPROACH THAT OF THIN FILM ELEMENTS AND NOT BE ANY MORE RELIABLE FROM THE STANDPOINT OF OVERLOAD CAPABILITIES.

A. SPECIFIC PROBLEM AREAS (CONT'D)

4. JUNGTION PROBLEMS:

MENTIONED EARLIER WERE THE DIFFICULTIES ENCOUNTERED WITH DIFFUSED AND EPITAXIAL ELEMENTS DUE TO THE P-N JUNCTION SURFACE EFFECTS. THE REVERSE CURRENT LEAKAGE, UNRELATED TO ZENER ÖRAVALANÇHE BREAKDOWN; IN A P-N JUNGTION CAN BE APPRE-CIABLE IF THE SURFACE OF THE SEMICONDUCTOR MATERIAL IS CONTAMINATED WITH RESIDUAL CHEMICALS (METALLIC SALTS, ETG.) WHICH ARE CON-DUCTIVE. IN THE MANUFACTURE OF DIODES, RECTIFIERS, ETC., EXTREME CARE MUST BE EXERCISED TO KEEP THE JUNCTION SURFACE CLEAN AND TO PASSIVATE THE SURFACE (BY THERMAL OXIDATION, FOR EXAMPLE) OF THE MATERIAL. THE PROBLEM OF INSTABILITY OF RESISTANCE, IN THE EVALUATION OF DIFFUSED ELEMENTS, WAS BELIEVED TO BE RELATED TO THE SENSITIVITY OF THE JUNGTION WHICH ISOLATES THE MORE HIGHLY DOPED LAYER FROM ITS SUBSTRATE. SURFACE PROTECTION WITH SILICONE VARNISH HELPED MINIMIZE THE CURRENT LEAKAGE AT HIGHER TEMPERATURES, BUT ROOM TEMPERATURE INSTABILITY REMAINED A PROBLEM.

OHMIC CONTACTS TO THE DIFFUSED AND EPITAXIAL ELEMENTS WERE ALSO COMPLICATED BY THE PRESENCE OF THE P-N JUNCTION. IN SOME CASES THE ALLOYED CONTACT WOULD PENETRATE THE LAYER TO THE SUBSTRATE AND THUS INTRODUCE A PARALLEL RESISTANCE WITH THE RESISTIVE LAYER ITSELF. ELEMENTS WHICH WERE FORMED BY VERY SHALLOW DIFFUSIONS WERE ESPECIALLY PRONE TO DO THIS.

THIS CONTACT PROBLEM DID NOT EXIST WITH ELEMENTS DEPOSITED

4. JUNCTION PROBLEMS (CONTID)

ON INSULATING SUBSTRATES.

5. SHIFTING TOR ON PACKAGING:

A PROBLEM OF NON-REPRODUCIBILITY OF TCR CHARACTERISTICS

BECAME GRITICAL IN THE LAST QUARTER. THE TCR UNENCAPSULATED

ELEMENTS WERE FOUND TO INCREASE AS MUCH AS 125 PPM/°C (AT 65°C

AND 175°C) AFTER MOLDING IN THE FINAL PACKAGE. THE INCREASE

IN RESISTANCE AND TCR WAS DUE TO A PIEZORESISTIVE EFFECT CAUSED

BY AN INDUCED STRESS IN THE SILICON ELEMENT. THE STRESS RE
SULTED FROM CONTRACTION OF THE EPOXY POTTING COMPOUND AS THE

DEVICE WAS COOLED FROM 200°C (FINAL CURE TEMPERATURE) TO ROOM

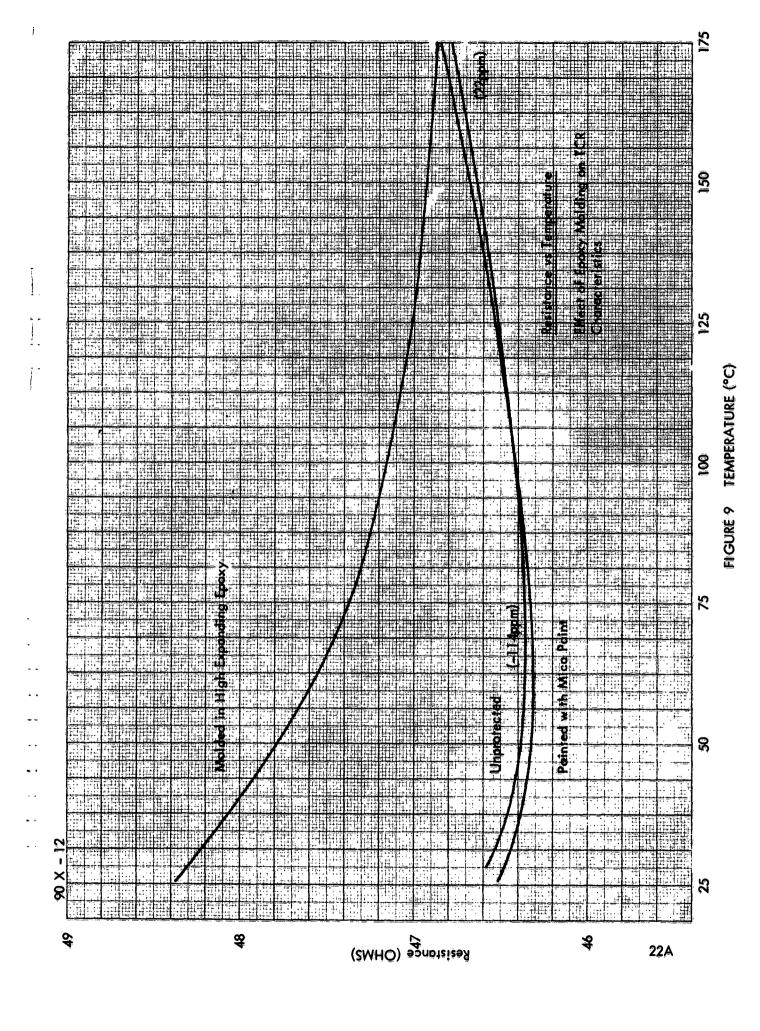
TEMPERATURE.

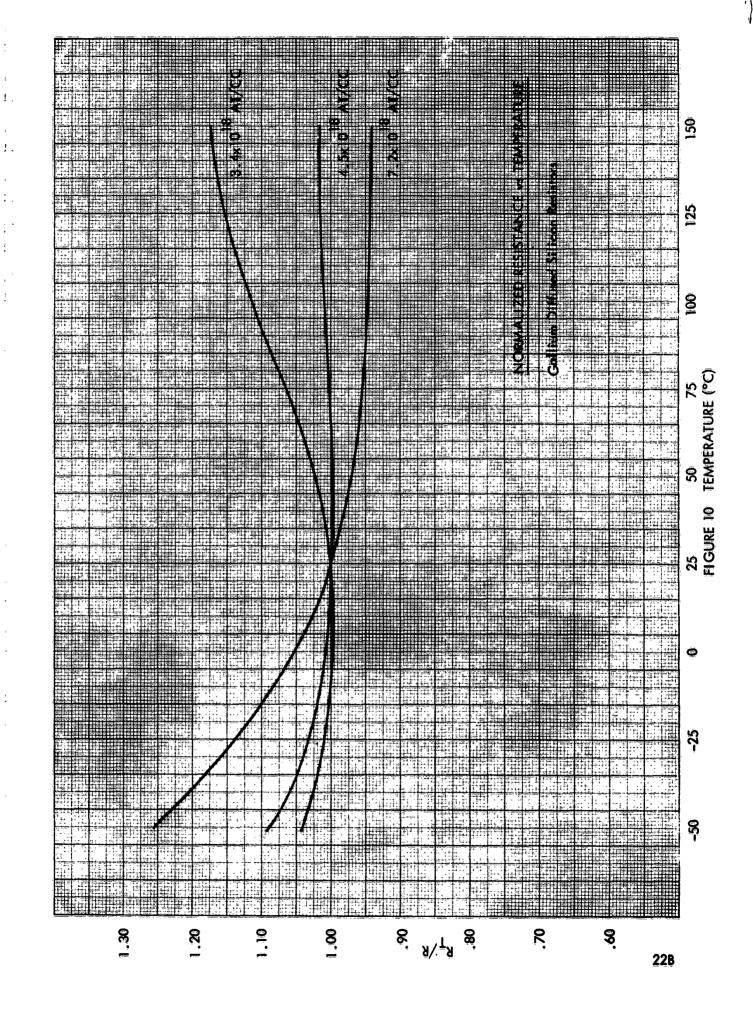
EVIDENCE OF THE PIEZORESISTIVE EFFECT IN THE SILICON ELEMENTS MAY BE SEEN FROM PLOTS OF RESISTANCE VERSUS TEMPÉRATURE BEFORE AND AFTER ENCAPSULATION. (SEE FIGURE 9) THE SOLUTION TO THE PROBLEM WAS TO USE A RESILIENT, LOWER SHRINKAGE, EPOXY AS THE FINAL ENCAPSULTION.

B. RESISTANCE -TEMPERATURE CHARACTERISTICS

1. DIFFUSED ELEMENTS:

Typical ReT curves for gallium diffeused elements of varying surface concentration may be seen in figure 10. The sheet resistance of these elements ranged from 50 to 100 Ω/sq .





1. DIFFUSED ELEMENTS (CONT'D)

JUNCTION DEPTH WAS APPROXIMATELY 0.3 MILS (.0003"). MINIMUM

TOR VALUES WERE OBSERVED AT GA CONCENTRATIONS OF APPROXIMATELY

5 x 10¹⁸ atoms/cc. A highly negative TOR below room temperature

WAS CHARACTERISTIC FOR ALL CONCENTRATIONS OF GA INVESTIGATED.

This low temperature behavior is a function of the activation

ENERGY OF THE MAJORITY CARRIERS IN THE SEMICONDUCTOR MATERIAL.

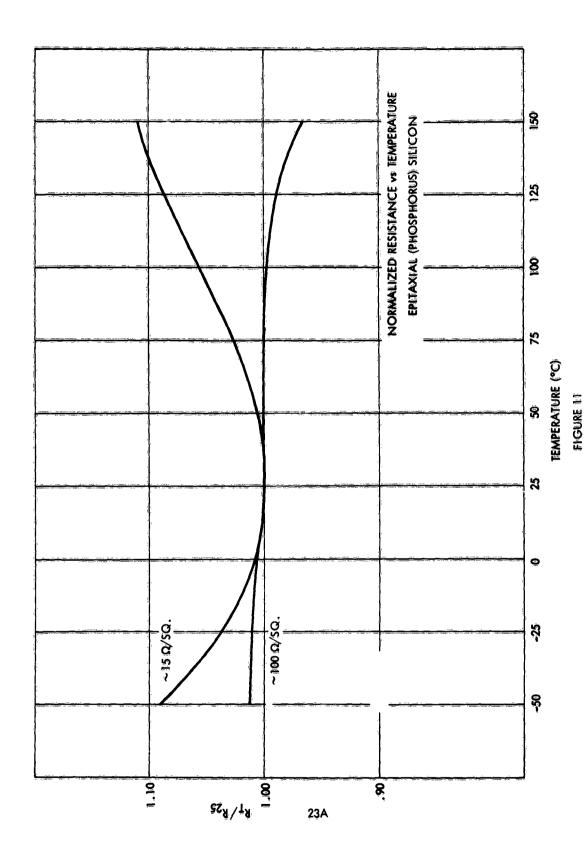
As the impurity atoms are ionized with increasing temperature,

THE RESISTIVITY DECREASES RAPIDLY FROM -50°C to 25°C.

THE RATE OF DECREASE IN RESISTIVITY WITH TEMPERATURE IN THIS LOW TEMPERATURE REGION IS PROPORTIONAL TO THE AMOUNT OF THERMAL ENERGY NEEDED TO COMPLETELY IONIZE THE CARRIERS - WHICH, IN TURN, IS A FUNCTION OF THE THERMAL ACTIVATION ENERGY OF PRE-DOMINANT IMPURITY ATOMS. GALLIUM HAS AN ACTIVATION ENERGY IN SILICON OF .065 EV.

2. EPITAXIAL ELEMENTS:

FIGURE 11 SHOWS RESISTANCE-TEMPERATURE CURVES FOR EPITAXIAL SILICON OF APPROXIMATELY THE SAME PHOSPHORUS DOPING LEVEL DEPOSITED AT TWO THICKNESSES. BY GROWING A VERY THIN (APPROXIMATELY 0.1 MIL, .0001") LAYER EPITAXIAL ELEMENT, THE SHEET RESISTANCE WAS INCREASED TO ABOUT $100 \Omega/\text{sq}$. The improvement in TCR characteristic is believed to be due to the high dislocation density at the interface of the thinner layer. In the thinner LAYER, MOST OF the CONDUCTION OCCURS IN THIS HIGH DISLOCATION



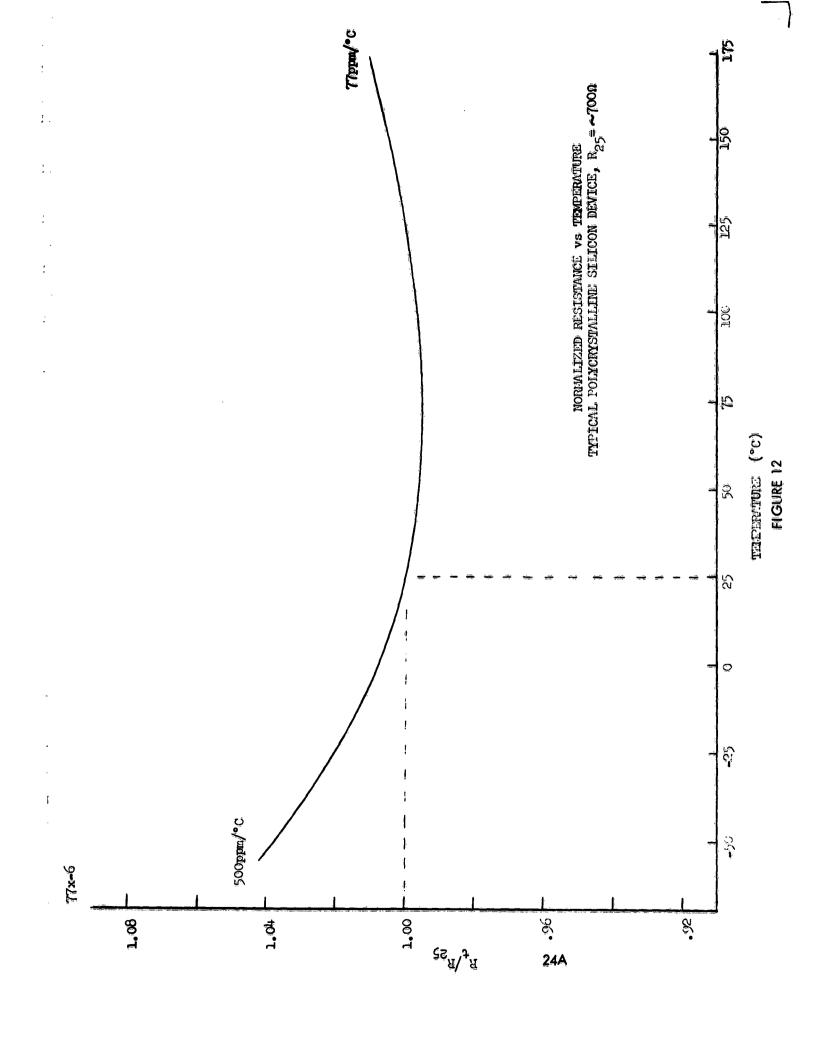
2. EPITAXIAL ELEMENTS (CONT'D)

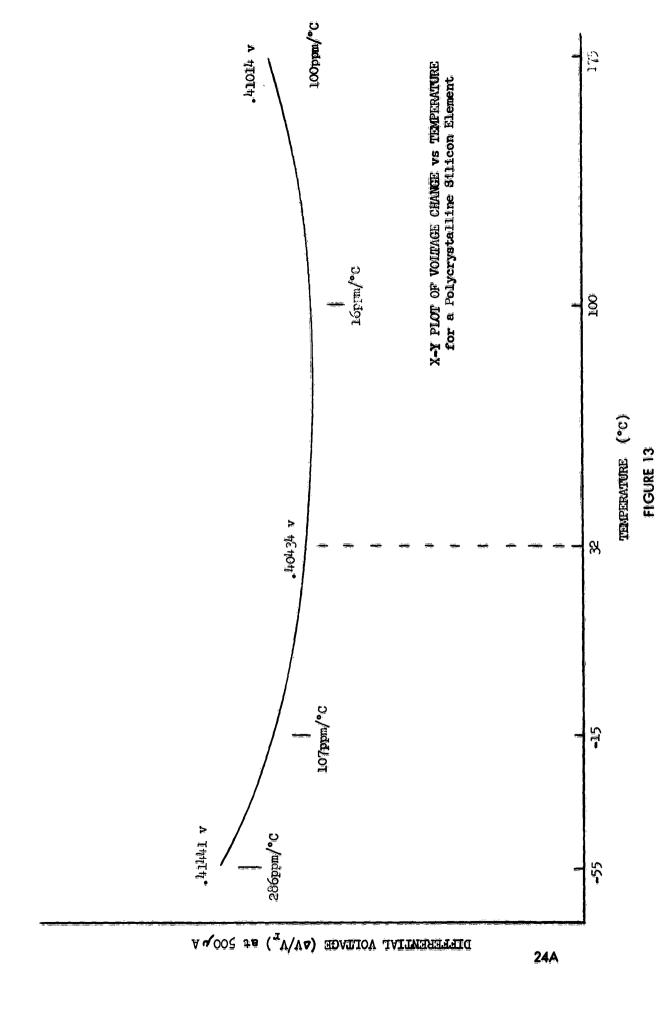
REGION AND THE CONDUCTION MECHANISM IS DETERMINED MAINLY BY
THE DISLOCATIONS. IN THE THICK LAYER MOST OF THE CONDUCTION
OCCURS AWAY FROM THE INTERFACE AND THE CONDUCTION MECHANISM,
WITH REFERENCE TO TEMPERATURE CHANGES, IS MORE TYPICAL OF
BULK ELEMENTS. AS STATED PREVIOUSLY, THE TEMPERATURE DEPENDENCE
OF THE JUNCTION LEAKAGE ALSO HAS AN EFFECT ON THE BEHAVIOR OF
THE RESISTANCE AT THE HIGHER TEMPERATURES.

IN THE COURSE OF THE WORK IT BECAME APPARENT THAT THE TCR
CHARACTERISTICS OF SINGLE CRYSTAL LAYERS WERE NOT SUFFICIENTLY
REPRODUCIBLE, NOR WERE TCR VALUES AS LOW AS COULD BE OBTAINED
WITH POLYCRYSTALLINE ELEMENTS.

3. POLYCRYSTALLINE ELEMENTS:

FIGURE 12 IS A TYPICAL CURVE FOR AN N-TYPE POLYGRYSTALLINE SILICON ELEMENT. MINIMUM TCR VALUES FOR POLYCRYSTALLINE ELEMENTS WERE OBTAINED AT DOPING LEVELS OF APPROXIMATELY 5 x 10¹⁹ ATOMS/CC OF PHOSPHORUS. FURTHER INCREASE IN DOPANT DID NOT IMPROVE THE OVER ALL CHARACTERISTICS OF THE TCR CURVE. THE SHAPE OF THE CURVE (NORMALIZED RESISTANCE VS TEMPERATURE) APPEARED TO BE AN INHERENT CHARACTERISTIC OF ALL THE POLY-CRYSTALLINE ELEMENTS EVALUATED. VARYING THE AMOUNT OF DOPANT IN THIS HIGHLY DOPED REGION (10¹⁹ TO 10²⁰ AT./CC) TENDED TO SHIFT THE MINIMUM POINT ON THE CURVE BUT DID NOT IMPROVE THE OVER ALL TCR CHARACTERISTICS. FIGURE 13 IS A CONTINUOUS PLOT





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3. POLYCRYSTALLINE ELEMENTS (CONT'D)

OF DIFFERENTIAL VOLTAGE VS TEMPERATURE FOR ONE OF THE POLY-CRYSTALLINE ELEMENTS.

A STUDY WAS MADE OF TOR AS A FUNCTION OF SMEET RESISTANCE.

SMEET RESISTANCE IS DEPENDENT UPON BOTH DOPING LEVEL (BULK
RESISTIVITY) AND THICKNESS.

THE TABLE BELOW LISTS THE SHEET RESISTANCE VALUES MEASURED ON A SERIES OF DEPOSITION RUNS AND SHOWS THE TCR OBTAINED FROM THE ELEMENTS. DEPOSITION COMDITIONS OF TEMPERATURE, CONCENTAINTION, AND TIME WERE HELD CONSTANT IN THESE RUNS.

SHEET RESISTANCE		C२
<u> </u>	AT 65°C	AT 175°C
16.9	- 42	÷171
14.9	+ 5 ⁸	+229
13.3	+ 65	+195
14.4	- 21	+161
17.5	- 53	+135
14.4	= 47	+13 8
17.6	+ 63	+206
13.2	+105	+237
18.6	-100	+ 62
15.8	+ 42	+230

There appears to be no correlation in sheet resistance within the limited Ω/sq values obtained and the TCR above room

3. POLYCRYSTALLINE ELEMENTS (CONT'D)

TEMPÉRATURE.

OF INTEREST WAS THE TCR OF A SINGLE CRYSTAL ELEMENT AND A POLYCRYSTALLINE ELEMENT DEPOSITED AT THE SAME TIME IN THE REACTOR. THE SINGLE CRYSTAL SLICE (ETCH-POLISHED SURFACE) WAS PLACED ADJACENT TO AN ALUMINA CERAMIC SUBSTRATE IN THE REACTOR AND SILICON WAS DEPOSITED. THE SHEET RESISTANCE OF THE SINGLE CRYSTAL ELEMENT WAS APPROXIMATELY ONE HALF THE VALUE OF THE POLYCRYSTALLINE ELEMENT. MEASURED RESISTANCE CHANGES WITH INCREASING TEMPERATURE SHOWED THAT THE SINGLE CRYSTAL ELEMENT HAD A HIGH POSITIVE TCR (APPROX 1200 PPM/°C AT 175°C) WHILE THE POLYCRYSTALLINE ELEMENT SHOWED A TYPICAL TCR VALUE FOR THIS DOPING LEVEL, I.E. APPROX 150 PPM/°C.

GRAIN BOUNDARIES IN POLYCRYSTALLINE MATERIAL WERE BELIEVED TO HAVE A SIGNIFICANT EFFECT ON THE ELECTRICAL BEHAVIOR OF THE RESISTIVE ELEMENTS. AT THE BOUNDARY OF EACH CRYSTALLITE, THERE WOULD BE A CONCENTRATION OF PHYSICAL DEFECTS (DISLOCATIONS) AS WELL AS A GREATER CONCENTRATION OF IMPURITY ATOMS. THE INTERFACE BETWEEN THIS MORE HIGHLY DOPED BOUNDARY AREA AND THE CRYSTALLITE ITSELF WOULD CONSTITUTE A POTENTIAL BARRIER.

THIS COULD EXPLAIN THE RELATIVELY GREATER SHEET RESISTANCE OF POLYCRYSTALLINE OVER SINGLE CRYSTAL LAYERS OF COMPARABLE DOPING LEVEL. THE TEMPERATURE DEPENDENCE OF THE CONDUCTIVITY WOULD BE A FUNCTION OF THE CRYSTALLITE, ITS GRAIN BOUNDARY,

3. POLYCRYSTALLINE ELEMENTS (CONT'D)

AND THE INTERFACE BETWEEN. THE POTENTIAL BARRIER ACROSS THE INTERFACES WOULD HAVE A TEMPERATURE DEPEDENT CONDUCTIVITY

SIMILAR TO A LOW BREAKDOWN DIODE. A MULTITUDE OF THESE

CRYSTALLITES AND INTERFACES RANDOMLY ORIENTED WOULD BEHAVE

ELECTRICALLY LIKE BACK-TO-BACK DIODES WITH LOW BREAKDOWN POTENTIALS

IN EITHER DIRECTION OF POLARITY. IT HAS BEEN THEORIZED THAT THE

EFFECTS OF THE GRAIN BOUNDARIES IN THE POLYCRYSTALLINE LAYERS

HAVE BEEN TO "SMOOTH OUT" THE RESISTANCE-TEMPERATURE CURVE BY

COMPENSATING TCR OF THE CRYSTALLITE WITH TCR OF THE BOUNDARY.

Two electron microscope photographs of the polycrystalline Layer structures (etched and as-deposited) which show grain structure have been included in the Supplementary Data Section. Also shown is a lapped cross-section through the silicon layer and alumina substrate. The penetration of the alumina ceramic by the pyrolytically deposited silicon, as observed in the micro photograph, provides an excellent bond between the resistive layer and its substrate.

III. DETAILED FACTUAL DATA (CONTINUED)

C. RELIABILITY INFORMATION:

THE FOLLOWING TABLE LISTS SOME OF THE MIL-R-10509D REQUIREMENTS AND THE ACTUAL RESULTS OBSERVED IN TESTING THE MOLDED POLY-CRYSTALLINE SILICON DEVICES.

TEST PERFORMED	MIL-R-10509D Requirement Char. C	ŘESULTS
RESISTANCE - TEMPERATURE CHARACTERISTIC	± 50 ррм (=55°C то 175°C)	Typical ±500 ppm at =55°C ±150 ppm at 175°C
Temperature Cycling	0.25%	.30 MAX .12 AVĜ
LOW-TEMPERATURE OPERATION	0.25%	.07 MAX .02 AVG
SHORT-TIME OVERLOAD	0.25%	.07 max .04 avg
OVERLOAD (5x) FOR 18 HOURS	-	1.0 MAX
Moisture Resistance (10 Day)	0.5%	.21 MAX .10 AVG
LOAD LIFE (1000 HOUR)	0.5%	.41, MAX .17 AVG (40 DEVICES TESTED AT 1/24)

DATA IN THIS SECTION INCLUDES:

1. LOAD LIFE DATA FOR 40 MOLDED DEVICES TESTED AT 125°C AND 1/2 WATT POWER FOR 1000 HOURS.



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•		424.29	424.59	+	-	424.89		-	70.52	C1 +	425,36		+ .25 425.22				
•		110 30 472 78	472	+ 0	=	472.93				7 . 16		10. +	+ .07 472.66				
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•		487.84	488,08	+	.05	418,33	1.10		488.58	+ .15	489.02 1 34	1.24	48.89	4.22			
'	AVERAGE																
	MEASURING EQUIPMENT:	MENT:															

NOTE: Resistances exceeding 1:00 Ohms are recorded as percent from nominal resistance.



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NOTE: Resistances exceeding 1100 Ohms are recorded as percent from nominal resistan .e.

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01-81	497.58	497.58 497.65	10. +	497.95		+ .07 498.58		. 20	+ .20 498.44			+ 1/2			
7-61	505.45	505.45 505.52	10.7	505.77	=	+ .06 505 82			30.905			111. +			
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PARAGRAIPH							4.6.13					A		4.6.3
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NOTE: Resistances exceeding 1100 Ohms are recorded as percent from nominal resistance.

REMARKS:



RESISTON TYPE		Mrs. DESIGNATION /2 (2 W		GROUP I	KESISTOR GUALITY ASSURANCE DATA SHEET GROUP IV TESTS, MIL-R-11050915	Y ASSURY EET WIL-R-1109	VNC.E	WITHE	WITHESSED BY DATE STARTED	DATE		Sheet John HUMBER	7 0
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PARAGRAPH							4.6.13						To the state of th	4.6.3
TEST		LOAD LIFE AT	7	125	ِ نِ	3	970	LOAD VOLTAGE	110		ř			SEAL
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5-610	773.02	773.02 773.66	,	773.82	01. +	773.76	4	773.92	7	773.93	4/- +			
7000	852.04	182.82	41	262.97	7	782.87	//	783.38		783.91	<i>7.1.</i> +			
309-4	838.81	839.12	+ 04	840.13	7/1	1 1/2 841.11	4 . 78	854.06	****************	853.60				
									-	076.70	**			
AVERAGE	789.29													

NOTE: Resistances exceeding 11:00 Ohms are recorded as percent from nominal resistance.

; ; DATE Transfer I make the second to TAKEN BY ٠<u>٨</u> (2.5 WATT LOAD for 18 HOLAS) POWER CONDITION DATA 1757° DATA SHËET (16-25)

REFUNE AFTER YOUR CALL TO SHIFT	1 526.30 S27.93	1705 28 508.46	114x 9 537.46 537.39 -, 61	115-x 6 551.06 551.50 + .09	114× 1 431.18 ミスル	93x11 7240672724 +. 43	108x 6 441.50 443.93 +.46	70×11 80,807 80813 4.0C	19 491.67 445.06 + .32	20 991.64 992.24 +,08	21 967.3596799 7.07	121 12- (20 650 34	85KIN 74.569 79.785 + . VE	85x1 76.58977.258 7.35	64x 2 83.670 83.816 +.17	92x 5 32.074 32.07401	18 1285,11265,0 - 01	69x10 55409 55444 +.07	108x11 844.39 840.84 43	5 505.16 903.64 13	2 855.60 853.67 -, 24	7 663.56 862.09 21	118x 1 772.51 771.53 -,13	115x 1 816.63 814.97 20	118x 14 711. 26 711.47 +.07
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74	74x3 177.77 678.59 +.19	34.70834.67010
¥ 1.9	81K3 619.16 617.63 20	57x c 685.82 (66.83 + 1.4
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72.	72x-4 (28,50,28.05 07	57.32837.410 1.2V
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X 6 9	28 18.381 14.041 2 xE8	54x 9 728.46 728.85 4.05
×2.5	- 77.657 15.857	54x10735.90 736 80 +.17
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ź,	12x 3 735,03737.31 +.31	57 X 11 775. 04 775.46 4.05
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A 63	K 3 750.60750.87 4.03	
	39. 35 236 40 24 . 18	

TEXAS INSTRUMENTS

PAGE OF LOG EVIDENCE OF LEAKAGE 1751-5 2-1541 SEAL 4.6.3 % RESISTANCE CHANGE TERMINAL STRENGTH 8-7-FINAL RESISTANCE READING 4.6.7 7 INITIAL RESISTANCE READING 1 WATHERSED BY 1 40. %. RESISTANCE 1.0.1 L 0 . 10.+ 1.07 0 1.03 ٠. ٢ 4.0 00. V= 3.4 1 624.06.627.81 311.88 311.74 45.8.04.7.78 304.16.363.99 417.41 417.97 413.30 423.20 FINAL RESISTANCE. 1 RESISTOR QUALITY ASSURANCE GROUP II TESTS, MIL-R-10509D INITIAL RESISTANCE READING DATA SHEET 0000 RESISTANCE 1.07 20. ÷ LOW TEMPERATIURE OPERATION $V = I \mathcal{U}$. نند ن ا ВĖ 311.74 424.69 462.65 289.84 264.74 72 729 INITIAL FINAL RESISTANCE RESISTANCE READING READING tr3.14 423.10 4.6.5 <u>!!</u> 3/1.74 417.74 623.81)) * RESISTANCE CHANGE Presy C. Rys TAL CINE TEMPERATURE CYCLING 287.67 FINAL RESISTANCE READING 423 29 PERSON PERFORMING TEST ALL Male & 623 6: 623.8 £ 2. **∥** ≪ 426 69 HHITHAL RESISTANCE READING 463 54 28-1-83 AVERAGE MARNENT: MEASUREMENT REQUIREMENT RES. READING PARAGRAPH STANDARD TEST WOLTAGE SAMPLE 28H

REMARKS:

SAMPLE CALCULATION

% Resistance Change= 100.(Final Resistance - Initial Resistance)

Initial Resistance = 39.590, Final Resistance = 39.526

% Resistance Change = 100(39.526 - 39.590) = -.161

For Resistance Volues Exceeding 1:00 Ohms, % Change ≈ (Final % of Nom. — Initial % of Nom.)

NOTE: Resistances exceeding 1100 Ohms are recorded as percent from nominal resistance.

!

Sheet 2 of 2 5-16 40 DAILY £ . 03 90 : -4 CHANGE 6 10.4 RESIS. ĸ +1 42.274 47.303 41.387 50.798 41.686 42.003 169.84 DRY RESIS. RE ADING FINAL ***** ŔΫ DATE COMPLETED 9-6-1 MEGOHMS. RESISTANCE INSULATION RESISTANCE **∦** 12-28-62 A RESIS. DIEL. WITHSTANDING VOLTAGE MOISTURE RESISTANCE WITHESSED BY DATE STARTED INIT. RESIS. FINAL RESIS. 4.6.11 READING *** READING RESISTOR QUALITY ASSURANCE GROUP III TESTS, MIL-R-10509D ₹ DATA SHEET - , 02 .02 20.-+ 07 + .01 Ŋ 10. 11. CHANGE 10. + RESIS. 41 48.639 48.666 43.021 41.954 47.843 47.344 42.220 45.215 42.990 42.993 WET RESIS. 41.345 41.336 READING 41.597 41.663 FINAL GNITLAL READING RESIS. ≡ Ķ CHANGE RESIS. EFIFECT OF SOLDERING alle 74 READING A 6.515. FINAL **∦ >** RESIS. INI THALL PERSON PERSONNE TEST ann MEASURING COUIPMENTS STANDARD RES. READING MEASUREMENT TI-2090 PARAGRAPH VOLTAGE AWERASE SAMPLE SPEC. REQ'T TEST TEST

NOTE: Resistances exceeding 100 Ohms are recorded as percent from nominal resistance.

BEMARKS:

C. RELIABILITY INFORMATION (CONTID)

1

- 2. RESULTS OF POWER CONDITIONING DEVICES AT 2.5 WATTS FOR 18 HOURS.
- 3. TEMPERATURE CYCLING, LOW-TEMPERATURE OPERATION, SHORT-
- 4. MOISTURE RESISTANCE DATA ON TEN MOLDED DEVICES.

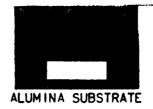
SEVERAL ELEMENTS TO WHICH SPECIAL LEADS WERE ATTACHED WERE OPERATED AT 20 WATTS FOR FIVE MINUTES DURATION WITH AN OBSERVED RESISTANCE SHIFT OF LESS THAN 0.5%. THE SILICON WAS UNPROTECTED FROM THE ATMOSPHERE. THIS TEST INDICATED THE INHERENT STABILITY OF THE SILICON ELEMENT WITH REGARD TO ELEVATED TEMPERATURE AND OXIDIZING AMBIENTS.

D. MANUFACTURING PROCEDURE:

A PROCESS AND MATERIAL FLOW CHART AND A PHOTOGRAPH OF A DISPLAY SHOWING STEPS IN FABRICATING THE POLYCRYSTALLINE SILICON DEVICE MAY BE SEEN ON THE FOLLOWING PAGES.

DETAILED MANUFACTURING SPECIFICATIONS FOR AN EPOXY MOLDED DEVICE HAVE BEEN OUTLINED IN THIS SECTION OF THE REPORT.

	FLOW Diagram	ĒA	MATERIAL		ÔPÉRAT I ÓN	FL Di	ÖW AĞRAM	ĒA	MATERIAL
PREPARATION OF SUBSTRATE	Q	1 x x x x x	ALUMINA SUBSTR TRICHLOROETHYL ACETONE DEIONIZED WATE NITRIC ACID SILICON TETRACHLORIO	È NË R	EPOXY UNDERGOATI TIN DIPPING	ING	\\ \\ \\ \\ \\ \\ \\ \\ \\ \\ \\ \\ \\	x x x	EPOXY PAINT (MICA FILLED) EPOXY PAINT THINNER SOLDER, PB/SN(70/30) SOLDER FLUX
VAPOR DEPOSITION	\ \ \ \ \ \ \ \ \ \ \ \ \ \ \ \ \ \ \	x x x x	PHOSPHOROUS TRICHLORIDE HYDROGEN HELIUM NITROGEN TRICHLOROETHYL	ĒNĒ	EPOXY CASTIN		~ \	X X X	EPOXY COMPOUND EPOXY HARDNER CODING INK INK THINNER
PREPARATION OF SILICON ELEMENT		X X X	HYDROFLUÖRIC A Deionized Wate Agetone						
VALUE ADJUSTMENT (PHOTO ETCH)	\$\frac{1}{2}\$	x x x x x	METAL ETGH RES PHOTO DEVELOPE SILICON ETCHAN TRICHLOROETHYL HYDROFLUORIG A DEIONIZED WATE AGETONE	R IT ENĒ IĒ IĐ					
APPLICATION OF OHMIC CONTAC	\\\\\\\\\\\\\\\\\\\\\\\\\\\\\\\\\\\\\\	X X X	ALUMINUM PÖWDE Copper powder Argon Argon/Hydrogen		:				
LEAD ATTACHMENT	V V V	X X X Z	SOLDER FLUX SOLDER, SN/AG DEIONIZED WATE ACETONE COPPER LEADS	Ŕ					
	<u> </u>	<u>ــلــ</u>		GKD GKD	Mark Verter	2	TEX	AS	S INSTRUMENTS
REVISIONS				APPD RELEASE	DATE		POXY	ŅÇ	ISTOR, APSULATION S & MATERIAL FLOW CHART)
-				N	EXT ASRY	вн 1 ов	1	A	NOBSR-85406



VAPOR DEPOSITED SILICON

K.M.E.R. APPLIED & DEVELOPED

VALUE ADJUSTED (PHOTO ETCHED)

OHMIC ALUMINUM CONTACT

COPPER OVERCOAT

LEAD ATTACH (SOLDER)

EPOXY UNDERCOAT

PREPARATION OF SUBSTRATE

EQUIPMENT

- 1. PYREX BEAKERS
- 2. GLASS STIRRING RODS
- 3. ULTRASONIC CLEANER
- 4. HOT PLATE
- 5. PLASTIC BEAKERS

MATERIALS

- 1. TRICHLORGETHYLENE
- 2. ACETONE
- 3. DEIONIZED WATER
- 4. NITRIC ACID
- 5. METHYL ALCOHOL
- 6. ALUMINA SUBSTRATES

PROCÉDURE

- 1. PLACE SUBSTRATES IN BEAKER OF METHYL ALCOHOL AND PUT IN ULTRASONIC CLEANER FOR A MINIMUM OF THIRTY (30) MINUTES.

 REPLACE METHYL ALCOHOL EVERY TEN (10) MINUTES UNTIL IT REMAINS CLEAN.
- 2. RINSE IN ACETONE.
- 3. WASH IN DEIONIZED WATER.
- 4. HEAT IN 50% SOLUTION OF NITRIC ACID UNTIL BOILING IS REACHED.

 REMOVE AND LET SET FOR FIVE (5) MINUTES. PUR OFF ACID.
- 5. RINSE IN DEPONIZED WATER.
- 6. STORE IN ACETONE UNTIL REQUIRED FOR DEPOSITION.

VAPOR DEPOSITION PROCEDURE

PRELIMINARY ADJUSTMENTS

(SEE FIGURE 14, VAPOR DEPOSITION SYSTEM)

- 1. CHECK VALVE "E". VALVE MUST BE IN POSITION NUMBER 1.
- 2. TURN FOUR-WAY VALVE "D" TO POSITION NUMBER 3.
- 3. TURN ON SWITCH "M" TO ACTIVATE EXHAUST FAN.
- 4. OPEN VALVE "L" AND IGNITE PILOT LIGHT.
- 5. ADJUST POWERSTAT "O" TO APPROXIMATELY 45%.

 NOTE: TEMPERATURE ON FLASH EVAPORATOR HEATING TAPE SHOULD

 BE APPROXIMATELY 50°C.
- 6. TURN ON VALVE "G", COOLING AIR.
- 7. TURN ON SWITCH "F", COOLING WATER.

 NOTE: CHECK DRAIN FOR PROPER FLOW.
- 8. TURN ON VALVES "I", "J", AND "K". ADJUST "K" TO APPROXIMATERLY 15 PSIG; ADJUST "J" TO 8 PSIG; ADJUST "I" TO 20 PSIG.

REACTOR OPERATING PROCEDURE

- 1. ADJUST FLOWRATERS "B", "C" TO INDICATE 13 SLM FLOW, HE.
- 2. ALLOW HE TO FLOW FIVE (5) MINUTES.

 NOTE: CHECK EXHAUST TO ASSURE UNIMPEDED FLOW OF GAS.
- 3. POSITION SUBSTRATES ON QUARTZ BOAT.
- 4. OPEN REACTOR AND PLACE BOAT ON ELECTRODE IN CENTER OF HEAT ZONE.
- 5. CLOSE REACTOR AND ALLOW HE TO FLOW ONE (1) MINUTE.
- 6. ADJUST POWERSTAT "N" TO CONTROL ELECTRODE TO APPROX 250°C.

 ALLOW HE TO CONTINUE FLOWING FOR FOUR (4) MINUTES MINIMUM.

- 7. TURN ON EMERGENCY SWITCH "H".
- 8. TURN VALVE "E" TO POSITION NUMBER 2.

 NOTE: EXHAUST MUST IGNITE WITHIN APPROX TWO (2) SECONDS.

 IF IT DOES NOT IGNITE, TURN VALVE "E" TO POSITION

 NUMBER 1 AND CHECK EQUIPMENT FOR GAS SUPPLY OR OBSTRUCTED

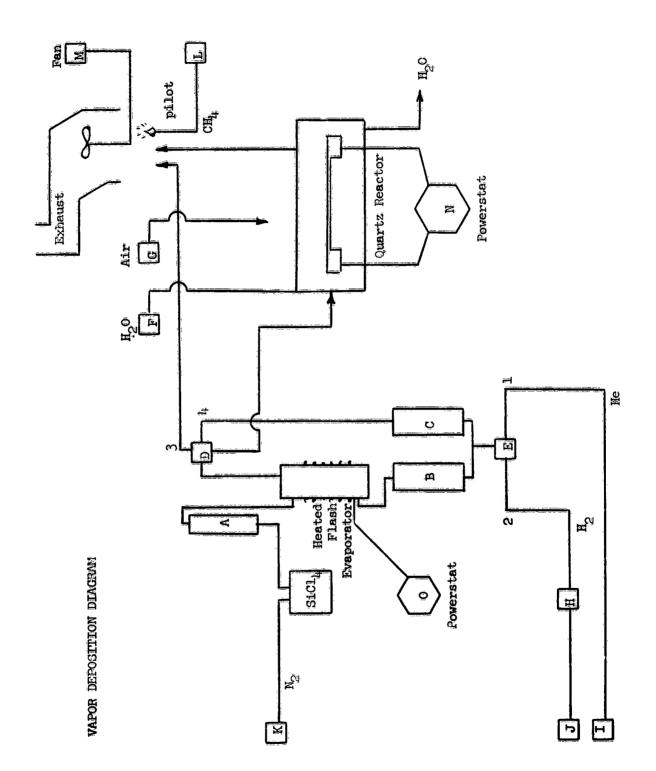
 EXHAUST LINE. WITH ASSURANCE OF PROPER EQUIPMENT SETUP,

 REPEAT STEP 8.
- 9. ADJUST FLOWRATERS "B" AND "C" TO INDICATE 15 SEM FLOW, Ha.
- 10. ADJUST POWERSTAT "N" TO CONTROL ELECTRODE TO APPROX 600°C.
- 11. ADJUST FLOWRATER "A" TO INDICATE 1.5 ML/MIN. FLOW, SIGLE (L).
- 12. ADJUST POWERSTAT "N" TO CONTROL ELECTRODE TO 1150°C ±10°C.
- 13. Turn four-way valve "D" to position number 4 starting peposition and allow to continue for defined time.
- 14. TURN VALVE "D" TO POSITION NUMBER 3 STOPPING DEPOSITION.
- 15. CLOSE FLOWRATER "A".
- 16. ALLOW ONE -HALF (1/2) MINUTE MINIMUM TO CLEAR SYSTEM.
- 17. ADJUST POWERSTAT "N" TO ZERO.
- 18. ALLOW ONE-HALF (1/2) MINUTE TO COOL.
- 19. TURN VALVE "E" TO POSITION NUMBER 1 AND ALLOW HE TO FLOW FOR FIVE (5) MINUTES.

PREPARATION OF SILICON ELEMENT

EQUIPMENT

- 1. PYREX BEAKERS
- 2. GLASS STIRRING RODS
- 3. PLASTIC BEAKER



MATERIALS

- 1. TRICHLORGETHYLENE
- 2. HYDROFLUORIC ACID
- 3. DE IONIZED WATER
- 4. ACETONE
- 5. VAPOR DEPOSITED SILICON ELEMENTS

PROCEDURE

- 1. RINSE IN ACETONE.
- 2. RINSE IN DEIONIZED WATER.
- 3. Wash IN 10% SOLUTION OF HYDROFLUORIC ACID FOR FIVE (5)
 MINUTES. POUR OFF ACID.
- 4. RINSE IN DETONIZED WATER.
- 5. STORE IN ACETONE UNTIL REQUIRED FOR OHMIC CONTACT
 APPLICATION.

VALUE ADJUSTMENT (PHOTO ETCH)

EQUIPMENT

- 1. CENTRIFUGAL SPINNER
- 2. DRYING OVEN CAPABLE OF MAINTAINING 120°C
- 3. LIGHT EXPOSURE TOWER
- 4. PHOTO PATTERN
- 5. HOT PLATE
- 6. PLASTIC BEAKERS
- 7. GLASS BEAKERS

MATER IALS

- 1. KODAK METAL ETCH RESIST (KMER)
- 2. PHOTO DEVELOPER
- 3. SILICON ETCHANT NUMBER 39E
- 4. TRICHLORGETHYLENE
- 5. HYDROFLUORIC ACID
- 6. DETONIZED WATER
- 7. ACETONE
- 8. METHYL ALCOHOL
- 9. PREPARED SILICON ELEMENTS

PROCEDURE

CAUTION: ALL KMER WORK MUST BE KEPT AWAY FROM DIRECT LIGHT

- 1. PLACE PREPARED SILICON ELEMENTS ON THE CENTRIFUGAL SPINNER (SILICON SIDE UP).
- 2. APPLY KMER IN DROPS ON THE ELEMENT (COVER ELEMENT COMPLETELY).
- 3. TURN SPINNER ON AND ALLOW THE EXCESS KMER TO BE THROWN OFF. A UNIFORM THIN LAYER IS DESIRED. DO NOT INCREASE SPINNER ACTION ENOUGH TO MOVE ELEMENTS.
- 4. Cure at ROOM TEMPERATURE FOR A MINIMUM OF TEN (10) MINUTES.
- 5. BAKE AT 120°C FOR TEN (10) MINUTES. ALLOW TO COOL.
- 6. PHOTO MASK WITH DESIRED PATTERN AND EXPOSE FOR 2-1/2
 MINUTES. MAKE SURE MASK IS HELD TIGHT AGAINST THE ELEMENT.

- D. MANUFACTURING PROCEDURE (CONT'D)
 - 7. SPRAY DEVELOPER ON ELEMENTS UNTIL PATTERN IS CLEAR.

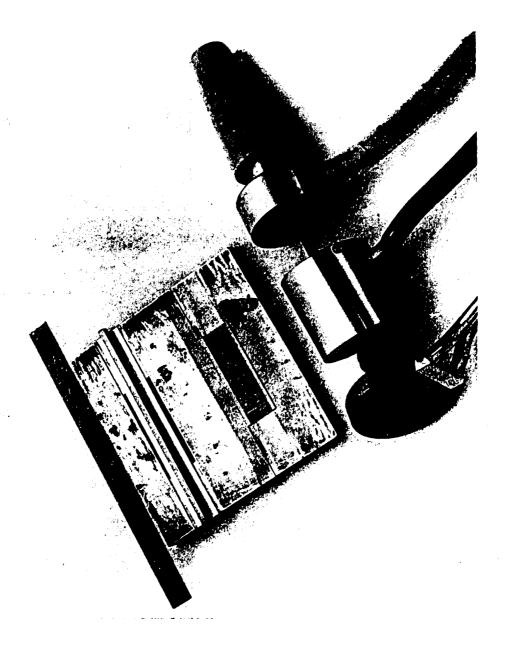
 DRY WITH AIR SPRAY TO ASSURE COMPLETE DEVELOPMENT.
 - 8. BAKE AT 120°C FOR TWENTY (20) MINUTES. ALLOW TO COOL.
 - 9. ETCH IN SILICON ETCHANT NUMBER 39E UNTIL PATTERN IS CLEAN AND SHARP. DO NOT PROLONG ETCHING. PROLONGED ETCHING WILL UNDERCUT KMER AND DESTROY PATTERN. POUR OFF ACID.
 - 10. RINSE IN DEIGNIZED WATER.
 - 11. CLEANING PROCESS:
 - 11.1 BOIL ELEMENTS IN TRICHLOROETHYLENE FIVE (5)
 MINUTES TO REMOVE KMER. MAKE SURE ALL KMER
 IS REMOVED.
 - 11.2 RINSE IN METHYL ALCOHOL.
 - 11.3 RINSE IN 10% SOLUTION HYDROFLUORIC ACID.

 (Use Plastic Beaker.) Pour off acid.
 - 11.4 BOLL IN DETONIZED WATER FIVE (5) MINUTES.
 - 12. STORE IN ACETONE UNTIL READY FOR APPLICATION OF OHMIC CONTACT.

APPLICATION OF OHMIC CONTACT

EQUIPMENT

- 1. MASKING JIG
- 2. PLASMA SPRAY GUN (PLASMADYNE CORPORATION, SANTA ANA, CALIFORNIA) (SEE FIGURE 15)



Plasma Spray Gun and Masking Jig FIGURE 15

MATERIALS

- 1. ALUMINUM POWDER (-325 MESH)
- 2. COPPER POWDER (-325 MESH)
- 3. ARGON
- 4. ARGON-HYDROGEN 95/5
- 5. PREPARED SILICON ELEMENTS

PROCEDURE

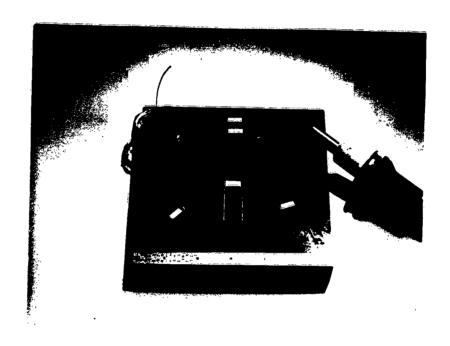
- 1. MOUNT SILICON ELEMENTS ON MASKING JIG.
- 2. PLACE A .500" METAL MASK ON THE ELEMENTS ALLOWING APPROXIMATELY .075" OF ENDS EXPOSED ON EACH SIDE.
- 3. SPRAY THE EXPOSED END WITH ALUMINUM POWDER USING THE MANUFACTURER'S INSTRUCTION FOR OPERATION OF THE PLASMA SPRAY GUN (SG-3). AMPERES SETTING OF 375.
- 4. SPRAY ALUMINUM CONTACT WITH AN OVERCOAT OF COPPER POWDER USING MANUFACTURING INSTRUCTIONS MENTIONED IN PARA. 3.

 AMPERES SETTING OF 250.
- 5. REMOVE ELEMENTS FROM JIG AND STORE IN PROPER CONTAINERS
 UNTIL REQUIRED FOR LEAD ATTACHMENT.

LEAD ATTACHMENT

EQUIPMENT

- 1. SOLDER IRON (40 WATT)
- 2. LEAD ATTACHMENT HOLDING JIG (SEE FIGURE 16)
- 3. CERAMIC PLATES



Lead Attachment Holding Jig FIGURE 16

MATERIALS

-

- 1. SOLDER SN/AG 95/5
- 2. SOLDER FLUX
- 3. DEIONIZED WATER
- 4. ACETONE
- 5. COPPER LEADS (OFHC)
- 6. OHMIG CONTACT APPLIED ELEMENTS

PROCEDURE

- 1. DIP END OF ELEMENT INTO SOLDER FLUX.
- 2. PLACE ON CERAMIC PLATE AND APPLY HOT SOLDERING IRON WITH EXCESS SOLDER ON TIP TO THE COPPER-COATED AREA. ALLOW THE SOLDER TO FLOW AND COVER THE OHMIC CONTACT AREA.
- 3. REPEAT STEPS (1) AND (2) FOR OPPOSITE END OF ELEMENT.
- 4. PLACE TINNED ELEMENT INTO HOLDING JIG.
- 5. DIP END OF LEAD WIRE IN SOLDER FLUX AND PLACE IN LEAD GROOVE ALLOWING THE FLUXED END TO EXTEND THE FULL LENGTH OF THE TINNED AREA.
- 6. APPLY HOT SOLDER IRON WITH EXCESS SOLDER ON TIP TO LEAD WIRE. ALLOW SOLDER TO FLOW AROUND WIRE AND COVER TINNED AREA. REMOVE SOLDERING IRON. DO NOT MOVE UNIT UNTIL SOLDER HAS SET.
- 7. REPEAT STEPS (5) AND (6) FOR OTHER END OF ELEMENT.
- 8. Wash ELEMENTS IN DEIGNIZED WATER TO REMOVE FLUX.
- 9. RINSE IN ACETONE AND ALLOW TO DRY.
- 10. STORE IN PROPER CONTAINER TO PREVENT CONTAMINATION UNTIL REQUIRED FOR EPOXY UNDERCOATING.

TIN DIPPING

EQUIPMENT

1. SOLDER POT CAPABLE OF MAINTAINING 200°C.

MATERIALS

- 1. SOLDER PB/SN 70/30
- 2. SUPERIOR FLUX NUMBER 30
- 3. TWEEZERS
- 4. METHYL ALCOHOL
- 5. EPOXY UNDERCOATED ELEMENTS

PROCEDURE

- 1. GRASP ELEMENT WITH TWEEZERS
- 2. DIP APPROXIMATELY 1/2 THE DISTANCE OF LEAD WIRE INTO
- 3. DIP FLUXED LEADS IN MOLTANT SOLDER UP TO THE PAINTED ELEMENT. DO NOT PROLONG TIME IN SOLDER POT. REMOVE FROM THE SOLDER AT A STEADY RATE.
- 4. Wash units in METHYL ALCOHOL TO REMOVE FLUX.
- 5. ALLOW TO DRY AND STORE IN PROPER CONTAINER UNTIL
 REQUIRED FOR EPOXY CASTING.

EPOXY UNDERCOATING

EQUIPMENT

1. PAINT BRUSH

MATERIALS

- 1. EPOXY PAINT (MICA FILLED) (2 PARTS PAINT:1 PART -13 MESH MICA BY WEIGHT)
- 2. EPOXY PAINT THUNNER (1:1 CELLOSOLVE ACETATE AND ISOPROPYL ALCOHOL = BE WEIGHT)
- 3. LEAD ATTACHED ELEMENTS

PROCEDURE

- 1. MIX PAINT THOROUGHLY UNTIL MICA IS WELL IN SUSPENSION.
- 2. APPLY A COAT OF PAINT TO THE SILICON ELEMENT SIDE OF THE SUBSTRATE. MAKE SURE THE CONTACT IS COMPLETELY GOVERED.
- 3. AIR DRY A MINIMUM OF THIRTY (30) MINUTES AT 38°C.
- 4. BAKE FIFTEEN (15) HOURS AT 200°C.
- 5. ALLOW UNITS TO COOL TO ROOM TEMPERATURE.
- 6. STORE UNITS UNTIL REQUIRED FOR TIN DIPPING.

MOLD ING

MANUFACTURER'S SPECIFICATIONS FOR CASTING A SEMIFLEXIBLE EPOXY MOLDING COMPOUND WERE FOLLOWED FOR FINAL ENCAPSULATION OF THE DEVICE. SILICONE RUBBER MOLDS WERE USED FOR BOTH IN-LINE AND RADIAL-LEAD CONFIGURATIONS.

IV. RECOMMENDATIONS

THE PYROLYTIC SILICON RESISTOR DEVELOPED UNDER THIS CONTRACT DID NOT COME UP TO EXPECTATIONS WITH RESPECT TO TCR (OVER THE TEMPERATURE RANGE OF -50°C TO +175°C). IN FACT, THE TCR IN THE LOW TEMPERATURE REGION WAS GREATER THAN THAT NORMALLY EXHIBITED BY CARBON-FILM RESISTORS. THE TCR ABOVE ROOM TEMPERATURE, AT 65°C AND 175°C, AVERAGED APPROXIMATELY 150 PPM/°C. TO SUBSTANTIALLY IMPROVE THESE TCR CHARACTERISTICS, I.E. FLATTEN THE R VS T CURVE, WOULD REQUIRE CONSIDERABLY MORE FUNDAMENTAL RESEARCH EFFORT THAN THAT EXPENDED IN THIS CONTRACT.

A POSSIBLE APPROACH TO BE CONSIDERED IN ANY FUTURE RESEARCH EFFORT WOULD BE TO DOPE THE SILICON WITH MORE THAN ONE IMPURITY OF THE SAME TYPE, E.G., PHOSPHORUS AND ARSENIC, IN HOPE OF MINIMIZING THE SLOPE OF THE RESISTIVITY CURVE IN THE LOW TEMPERATURE REGION. THIS, IN EFFECT, MIGHT BE EQUIVALENT TO DOPING WITH AN IMPURITY OF VARYING ACTIVATION ENERGY. SUCH A PROGRAM WOULD BE VERY EXTENSIVE AND HAD TO BE CONSIDERED OUTSIDE THE REALM OF THIS CONTRACT. JUSTIFICATION OF SUCH AN EFFORT WOULD REST MAINLY UPON THE ADDITIONAL ADVANTAGES (OTHER THAN TCR) WHICH MIGHT ENSUE. ONE OF THE ADVANTAGES ALREADY DEMONSTRATED WITH THE PRESENT DEVICE IS ITS RESISTANCE TO OXIDATION AND HIGH TEMPERATURE CAPABILITIES. EVEN SO, CONTINUATION OF THE R&D EFFORT TO REDUCE THE TCR OF THE SILICON DEVICE IS NOT ADVISED. NEITHER IS A MANUFACTURING-METHODS TYPE CONTRACT RECOMMENDED FOR EXPLOITING THE PROCESS THUS FAR EVOLVED.

IV. RECOMMENDATIONS (CONTINUED)

IN SPITE OF THE INABILITY TO MEET THE CONTRACT OBJECTIVES WITH THE SILICON LAYER RESISTIVE ELEMENT, SOME THOUGHT WAS GIVEN TO PILOT-LINE OR COMMERCIAL-SCALE PRODUCTION. PYROLYTIC DEPOSITION OF SILICON BY HYDROGEN REDUCTION OF THE HALIDE IS A WIDELY USED PROCESS IN THE SEMICONDUCTOR INDUSTRY, AND CONTINUOUS DEPOSITION SYSTEMS HAVE BEEN PROPOSED.

THE SILICON RESISTOR AT THIS STAGE OF ITS DEVELOPMENT HAS CERTAIN PROPERTIES WHICH SUGGEST POTENTIAL USES. ONE OF THESE IS STABILITY AT ELEVATED TEMPERATURES. IT WAS RECOMMENDED THAT A PYROLYTIC (POLYCRYSTALLINE) SILICON DEVICE UNPROTECTED FROM THE ATMOSPHERE WOULD OPERATE AT POWER LEVELS SUFFICIENT TO RAISE ITS TEMPERATURE TO RED HEAT. ONLY A SMALL CHANGE IN RESISTANCE (~1.0%) RESULTED FROM THIS TREATMENT. THIS WOULD SUGGEST THE POSSIBILITIES OF THE SILICON RESISTIVE ELEMENT FOR HIGH-TEMPERATURE DEVICES.

FINALLY, THE HIGH-TEMPERATURE CAPABILITIES OF THE DEVICES WOULD REQUIRE

A PACKAGE WHICH COULD WITHSTAND THE ABUSE OF EXCESSIVE HEAT AND

OXIDATION. THE PACKAGING MATERIAL SHOULD ALSO PROVIDE GOOD HEAT

DISSIPATION FOR THE ELEMENT. COMBINATIONS OF CERAMICS AND GLASS COULD

BE DESIGNED TO PROVIDE A FINAL ENCAPSULATION FOR THIS DEVICE.

PACKAGES SIMILAR TO THOSE PRESENTLY USED FOR POWER RECTIFIERS MIGHT ALSO BE APPLICABLE.

SUPPLEMENTARY DATA

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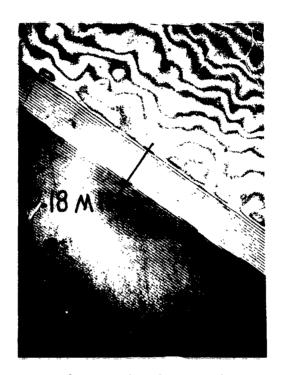
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Optimize conditions of vapor-deposition process for controlable runs Adjust resistance by electron beam scribing and photo-resist ētching Improve ohmic contacts for greater mechanical strength and power dissipation Determine effect of substrate resistivity SCHEDULE AND PERFORMANCE FOR PHASE II on TCR of epitaxial layer elements BUSHIPS CONTRACT NOBSE 85406 increase sheet resistivity of diffused and SEMICONDUCTOR RESISTIVE ELEMENT vapor-deposited é l'émen ts Investigate effect of thermal history and layer thickness on TCR of vapor-dep elements Design a package for encapsulation of elements for environmentāl testing Lower TCR of elements to meet contract objective performance (or request change in specs) Fabricate and test a sufficient number of finished devices for reliability information Build, test and submit 20 of best (lowest TCR) most stable, etc.) ele ments to BuShips Evaluate and furnish 150 engineering samples of low TCR devices to BuShips Complete final engineering report

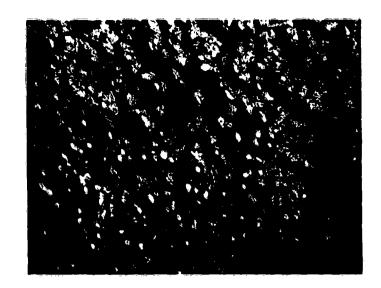
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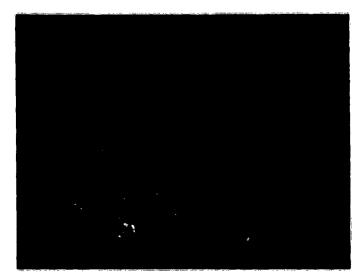
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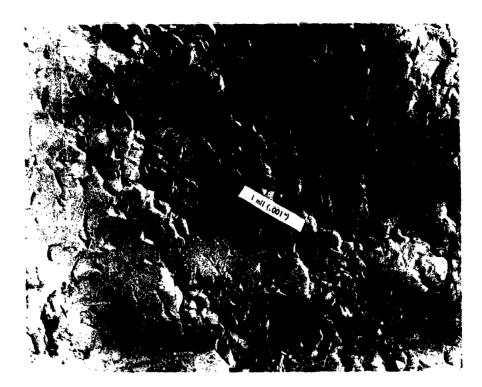
A. Micro Photograph of Epitaxial Silicon Layer, Stained and Viewed under Sodium Light



Polycrystalline Silicon 1000x



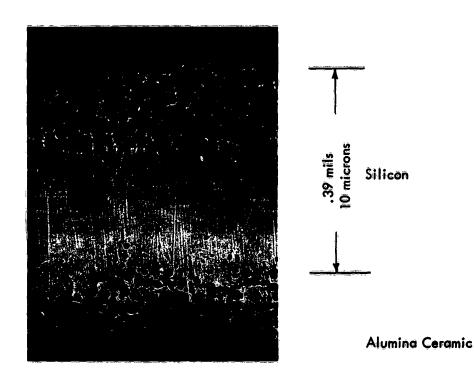
Etched Alumina Substrate 1000×



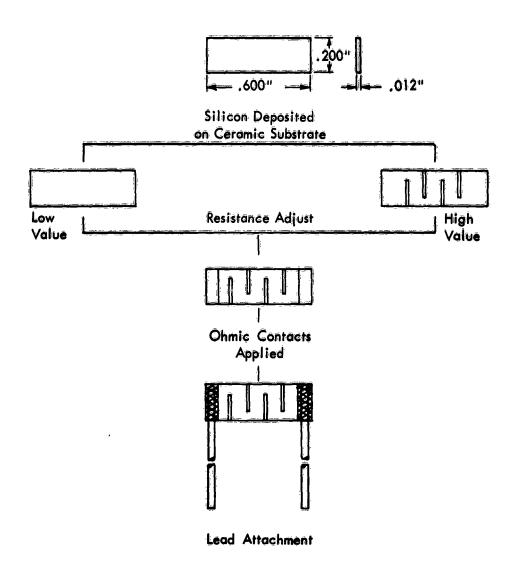
Polycrystalline Silicon (6000×) As - Deposited Surface



Polycrystalline Silicon (6000x) Etched Surface



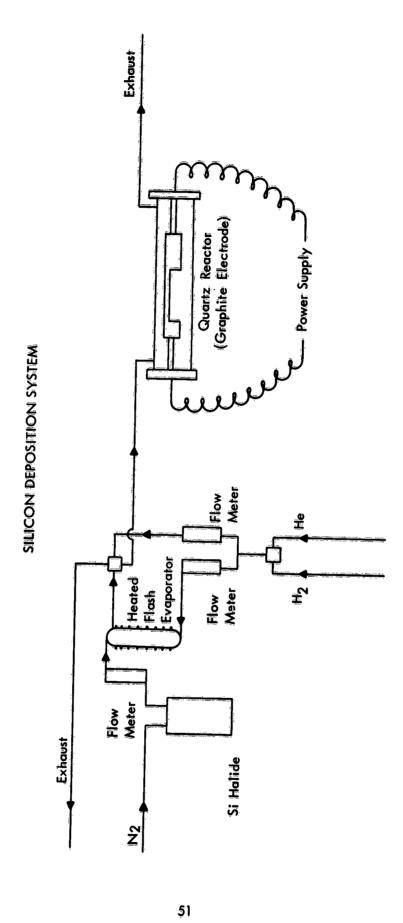
Lapped Cross-Section Through Silicon Layer - Alumina Substrate (400x)



.800" TI .350"

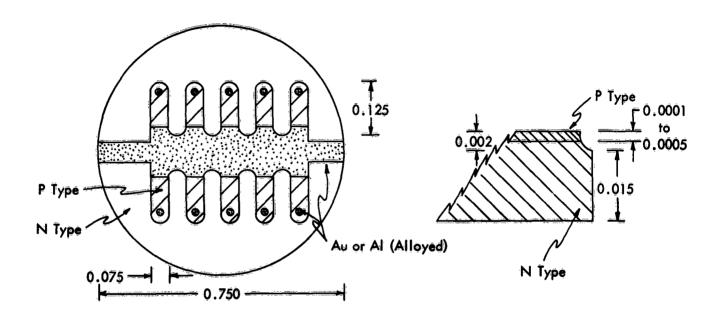
ASSEMBLY AND PACKAGING FLOW DIAGRAM

Encapsulation

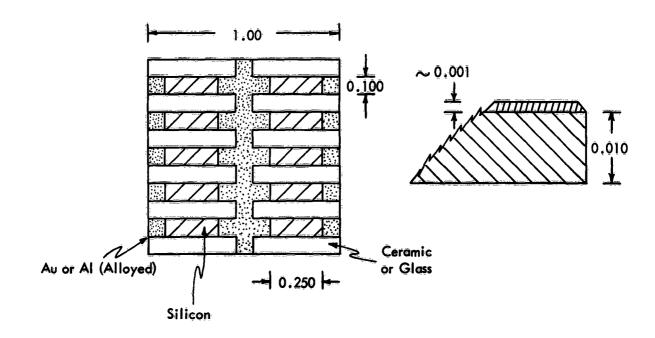


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CONFIGURATION FOR EPITAXIAL AND DIFFUSED SILICON ELEMENTS



CONFIGURATION FOR POLYCRYSTALLINE ELEMENTS



55. DATA SHEET (16-25)

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Sheet 2 of 2 subsections S = 1609	PAGE OF DAILY LOG	6.6.3	SEAL	EVIDENCE	LEAKAGE									
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NOTE: Resistances exceeding 3100 Ohms are recorded as percent from nominal resistance.

AVERAGE MEASURING EQUIPMENT:

REMARKS:

TEXAS INSTRUMENTS
IN COMPONENTS DIVISION
CONTOUNTS DALLASS STEAM

RESISTOR QUALITY ASSURANCE GROUP IV TESTS, MIL-R-10509D DATA SHEET

38

MPG. DESIGNATION

TI-1967

DANCY 5-16 09 Sheet 2 of 2 John Now Now No. 1 ġ Ú ľ DATE COMPLETED 12-26 11-13-62 DATE STARTED WITHESSED BY

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NOTE: Resistances exceeding 1100 Ohms are recoided as percent from nominal resistance.

SIGNATURES

THIS CONCLUDES THE FINAL ENGINEERING REPORT FOR SEMICONDUCTOR RESISTIVE ELEMENTS UNDER CONTRACT NOBSR-85406.

RODGER B. HERRINGTON

OPERATIONS CONTRACT MANAGER

OLIN B. CECIL

MANAGER, PRODUCT ENGINEERING BRANCH

RESISTOR DEPARTMENT

RAYMOND D. PUCKETT

MANAGER, RESISTOR DEPARTMENT